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GUIDELINES FOR DEVELOPMENT OF A QUALITY ASSURANCE PROGRAM

VOLUME III. DETERMINATION OF MOISTURE IN STACK GASES

RESEARCH TRIANGLE INSTITUTE

PREPARED FOR
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1. Good operating practices
2. Directions on how to assess performance and qualify data
3. Directions on how to identify trouble and improve data quality
4. Directions to permit design of auditing activities

The document is not a research report. It is designed for use by operating personnel.

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GUIDELINES FOR DEVELOPMENT OF A QUALITY ASSURANCE PROGRAM: VOLUME III - DETERMINATION OF MOISTURE IN STACK GASES

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ABSTRACT

Guidelines for the quality control of determination of moisture in stack gases by the Federal reference method are presented. These include:

1. Good operating practices
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This work was submitted in partial fulfillment of Contract Durham 68-02-1234 by Research Triangle Institute under the sponsorship of the Environmental Protection Agency. Work was completed as of December 1974.

SECTION I INTRODUCTION

This document presents guidelines for developing a quality assurance program for Method 4, Determination of Moisture in Stack Gases. This method was published by the Environmental Protection Agency in the Federal Register, December 23, 1971, and is reproduced as appendix A of this report for convenience of reference.

This document is divided into four sections:

Section I, Introduction. The Introduction lists the overall objectives of a quality assurance program and delineates the program components necessary to accomplish the given objectives.

Section II, Operations Manual. This manual sets forth recommended operating procedures to insure the collection of data of high quality, and instructions for performing quality control checks designed to give an indication or warning that invalid data or data of poor quality are being collected, allowing for corrective action to be taken before future measurements are made.

Section III, Manual for Field Team Supervisor. This manual contains directions for assessing data quality on an intralaboratory basis and for collecting the information necessary to detect and/or identify trouble.

Section IV, Manual for Manager of Groups of Field Teams. This manual presents information relative to the test method (a functional analysis to identify the important operations variables and factors, and statistical properties of and procedures for carrying out auditing procedures for an independent assessment of data quality.

The objectives of this quality assurance program for Method 4 are to:

1. Minimize systematic errors (biases) and control random variability (precision) within acceptable limits in the measurement process,
2. Provide routine indications for operating purposes of satisfactory performance of personnel and/or equipment,
3. Provide for prompt detection and correction of conditions that contribute to the collection of poor quality data, and
4. Collect and supply information necessary to describe the quality of the data.

To accomplish the above objectives, a quality assurance program must contain the following components:

1. Recommended operating procedures,
2. Routine training of personnel and evaluation of performance of personnel and equipment,
3. Routine monitoring of the variables and parameters that may have a significant effect on data quality,
4. Development of statements and evidence to qualify data and detect defects, and
5. Action strategies to increase the level of precision/accuracy in the reported data.

Component (2) above will be treated for all the methods in the final report of this contract. Component (5) is treated in the Quality Assurance Documents for pollutant specific methods requiring the results of Method 4.

Implementation of a properly designed quality assurance program should enable measurement teams to achieve and maintain an acceptable level of precision and accuracy in their stack gas composition measurements. It will also allow a team to report an estimate of the precision of its measurements for each source emissions test.

Variability in emission data derived from multiple tests conducted at different times includes components of variation from:

1. Process conditions,
2. Equipment and personnel variation in field procedures, and
3. Equipment and personnel variation in the laboratory.

In many instances time variations in source output may be the most significant factor in the total variability. The error resulting from this component of variation is minimized by knowing the time characteristics of the source output and collecting the gas sample at a rate proportional to the stack gas velocity. The sampling period should span at least one complete output cycle when possible. If the cycle is too long, either the sample collection should be made during a portion of the cycle representtative of the cycle average, or multiple samples should be collected and averaged.

Quality assurance guidelines for Method 4 as presented here are designed to insure the collection of data of acceptable quality by prevention, detection, and quantification of equipment and personnel variations in both the field and the laboratory through:

1. Recommended operating procedures as a preventive measure,

2. Quality control checks for rapid detection of undesirable performance, and

3. A quality audit to independently verify the quality of the data.

The scope of this document has been purposely limited to that of a field and laboratory document. Additional background information is contained in the final report under this contract.

SECTION II OPERATION'S MANUAL

2.0 GENERAL

This manual sets forth recommended procedures for the determination of stack gas moisture according to Method 4 (Method 4 is reproduced from the Federal Register and is included as appendix A of this document). Quality control procedures and checks designed to give an indication or warning that invalid or poor quality data are being collected are written as part of the operating procedures and are to be performed by the operator on a routine basis. In addition, the performance of special quality control procedures and/or checks as prescribed by the supervisor for assurance of data quality may be required of the operator on special occasions.

The sequence of operations to be performed for the measurement process is given in figure 1. Each operation or step in the method is identified by a block. Quality checkpoints in the measurement process, for which appropriate quality control limits are assigned, are represented by blocks enclosed by heavy lines. Other quality checkpoints involve go/no-go checks and/or subjective judgments by the test team members with proper guidelines for decisionmaking spelled out in the procedures.

The precision/accuracy of data obtained from this method depends upon equipment performance and the proficiency with which the operator performs his various tasks. From equipment calibration through on-site measurements, calculations, and data presentation, this method is susceptible to a variety of errors. Detailed instructions are given for minimizing or controlling equipment error, and procedures designed to minimize personnel errors are recommended. Before using this document the operator should study Method 4 as written in appendix A in detail.

For discussion purposes, the measurement process is divided into three phases:

1. Apparatus selection,
2. Presampling preparation, and
3. On-site measurements.

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APPARATUS SELECTION

1. SELECT THE APPARATUS ACCORDING TO SPECIFICATIONS GIVEN FOR THE REFERENCE METHOD (SECTION 2, APPENDIX A) AND TO SUBSECTION 2.1.

PRESAMPLING PREPARATION

2. PERFORM VISUAL AND OPERATIONAL CHECKS OF EQUIPMENT ACCORDING TO SUBSECTION 2.2.1.

3. CALIBRATE THE DRY GAS METER, ROTAMETER, AND BAROMETER ACCORDING TO SUBSECTION 2.2.2.

4. PACKAGE EQUIPMENT FOR SHIPMENT TO THE FIELD SITE (SUBSECTION 2.2.3).

ON-SITE MEASUREMENTS

5. ASSEMBLE AND CHECK EQUIPMENT FOR PROPER OPERATION ACCORDING TO SUBSECTION 2.3.2.

6. COLLECT SAMPLE ACCORDING TO SUBSECTION 2.3.3.

7. MAKE VOLUMETRIC (OR GRAVIMETRIC) MEASUREMENTS TO DETERMINE VOLUME OF WATER COLLECTED.

8. PERFORM CALCULATIONS TO DETERMINE MOISTURE CONTENT ACCORDING TO SUBSECTION 2.3.4.

9. VALIDATE DATA BY COMPARING THE MEASURED VALUE TO THE THEORETICAL VALUE CALCULATED FROM COMBUSTION NOMOGRAPHS USING PROCESS DATA.

10. PACK EQUIPMENT IN ORIGINAL CONTAINERS FOR SHIPMENT TO THE HOME LABORATORY.

POSTSAMPLING OPERATIONS

11. FORWARD DATA FOR ADDITIONAL INTERNAL REVIEW OR TO USER.

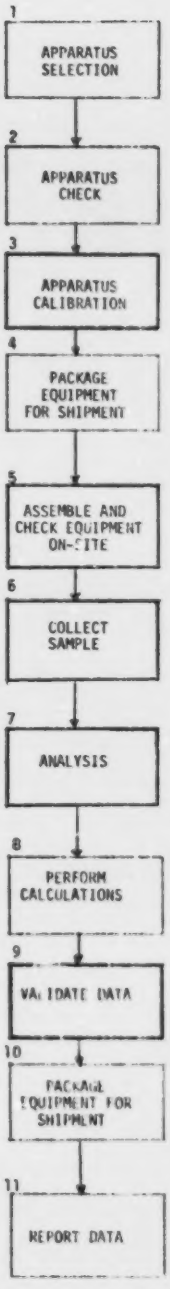

Figure 1. Operational flow chart of the measurement process.

2.1 APPARATUS SELECTION

A listing of the required apparatus with certain pertinent specifications is given in section 2 of appendix A. Also, a schematic of an assembled moisture-sampling train with all components identified is shown in figure 4-1 of appendix A. Additional specifications, criteria, and/or design features as applicable are given here to aid in the procurement of equipment to insure the collection of data of acceptable quality. Procedures and limits for acceptance checks of new equipment are given. A descriptive title and the identification number of new equipment should be recorded in a receiving record file. The entry should be dated and signed by the individual who performed the acceptance check. Calibration data obtained as a part of the acceptance check should be recorded in the calibration log book.

2.1.1 Sampling Probe

2.1.1.1 Design Characteristics. A sampling probe constructed of stainless steel or borosilicate (Pyrex) glass is suggested. The probe tip should have provision for retention of a particulate filter. It should have a suitable connection for making a leak-free connection with the condenser unit. The probe must have a heating system capable of maintaining a temperature sufficient to prevent condensation in the probe while sampling.

For ease of cleaning the inside diameter of the probe should not be less than about 1/4 inch. For structural stability and ruggedness, a glass probe should have a wall thickness of at least 1/16 inch.

2.1.1.2 Acceptance Check. When first received, a probe should be visually checked for any signs of damage. The probe heating system should be checked by assembling the sampling train as in figure 4-1 of appendix A, but without the two impingers.

1. Connect the probe (without filter) to the inlet of the pump.
2. Electrically connect and turn on the probe heater for 2 or 3 minutes. It should become warm to the touch over the entire length of the probe.
3. Start the pump and adjust the needle valve until a flow rate of about 0.075 ft^3/min is achieved.
4. The probe should remain warm to the touch over its entire length.

The heater should be capable of maintaining a minimum temperature of 250°F under these conditions. If it cannot, the probe should be rejected.

2.1.2 Drying Tube

A drying tube (glass or plastic) with a minimum capacity of 30 grams of silica gel (6-16 mesh, grade 42 indicating type) or a third impinger filled with silica gel should be used in the sampling train to protect the pump and dry gas meter against excess moisture and to serve as an indication of the collection efficiency of the impingers.

2.1.3 Pitot Tube and Differential Pressure Gage

The Quality Assurance Document of this series for Method 2 should be consulted for the maintenance and use of the type-S pitot tube and differential pressure gage.

2.1.4 Impingers

2.1.4.1 Design Characteristics. Two 25-ml midget impingers with suitable connections are required.

2.1.4.2 Acceptance Check. Each impinger is checked visually for damage, such as breaks or cracks, and manufacturing flaws, such as poor fitting connections. The impingers are accepted if no damage or flaws are detected.

2.1.5 Dry Gas Meter

2.1.5.1 Design Characteristics. A dry gas meter operable at flow rates from about 0.05 to 0.1 ft^3/min with 2 percent accuracy for total volume is required. A rating of 0.1 ft^3/ revolution is recommended.

2.1.5.2 Acceptance Check. The manufacturer of commercial sampling trains should provide a calibration curve over the expected operating range. The dry gas meter should be visually checked for damage, then set up, and a three-point calibration check performed as described in subsection 2.2.2.1. If γ at either one of the three check points falls outside the limits of 1.0 ± 0.02, the meter should be adjusted and recalibrated, recalibrated and a calibration curve constructed, or rejected.

2.1.6 Pump

2.1.6.1 Design Characteristics. The vacuum pump must be leak-free. It

should be operable at 120 volts, 60 Hz, and equipped with a 3-wire electrical cord to insure proper grounding. A pump capable of pumping 0.1 ft^3/min at 20 inches of mercury vacuum is acceptable.

2.1.6.2 Acceptance Check. Make a performance check to verify that the pump satisfies the manufacturer's specifications. If the pump leaks or does not meet the stated specifications, it should be rejected.

2.1.7 Rotameter

2.1.7.1 Design Characteristics. The rotameter should have a range of 0 to 0.1 ft^3/min.

2.1.7.2 Acceptance Check. A calibration curve is to be supplied by the manufacturer. The rotameter is checked against the calibrated dry gas meter with which it is to be used as directed in subsection 2.2.1.3. If the rotameter is not within $\pm$ 5 percent of the manufacturer's calibration curve, recalibrate and construct a new calibration curve. This procedure would also correct for a barometric pressure that differs significantly from standard pressure at which the manufacturer's calibration was made.

2.1.8 Barometer

2.1.8.1 Design Characteristics. The barometer, usually an aneroid barometer, should be capable of measuring atmospheric pressure to within 0.1 inches of Hg.

2.1.8.2 Acceptance Check. Check the field barometer against a mercury-in-glass barometer or equivalent. Adjust the field barometer to agree with the mercury barometer if they differ by more than 0.2 inches of Hg. Reject the barometer if it cannot be adjusted or shows erratic behavior.

2.1.8.3 Documentation. Record in the receiving record book a description of the barometer, its serial number, and results of the acceptance check. Date and sign the entry.

2.1.9 Graduated Cylinder

A 25-ml graduated cylinder with 0.2-ml divisions is recommended for volumetric measurements. The cylinder is visually checked for damage and manufacturing flaws. Reject faulty cylinders.

2.1.10 Needle Valve

A metering valve with a conveniently sized fitting is required. With the sampling train as shown in figure 4-1 of appendix A, the metering valve cannot be an integral part of the rotameter. Visually check the needle valve for damage. Document receiving the valve in the receiving record book.

2.2 PRESAMPLING PREPARATION

2.2.1 Apparatus Check

Each item in the sampling train should be visually checked for damage and/or excessive wear before each field test. Items should be repaired or replaced as applicable if judged to be unsuitable for use by the visual inspection.

Table 1 is a sample equipment check list and is designed to serve as a check list for the three phases of a field test. It is meant to serve as an aid to the individuals concerned with procuring and checking the required equipment, and as a means for readily determining the equipment status at any time. The completed form should be dated, signed by the field crew supervisor, and filed in the operational log book upon completion of a field test. This includes initiating the replacement of worn or damaged items of equipment. Procedures for performing the checks are given in the appropriate subsections of this operations manual. A check is placed in the proper row and column of table 1 as the check/operation is completed.

In addition to a visual check, the following performance and/or calibration checks are performed before each field test.

2.2.1.1 Sampling Train Leak Check. Assemble the sampling train as shown in figure 4-1 of appendix A. Without the particulate filter in the probe and with no water in the impingers, leak-check the sampling train by plugging the probe inlet and pulling a vacuum by letting the pump run. Leaks greater than 1 percent of the sampling rate (i.e., about 0.00075 ft^3/min) as indicated by the dry gas meter, should be found and corrected before continuing.

2.2.1.2 Dry Gas Meter Calibration Check. After the leak check has been

Table 1. Moisture measurement check list

TEST SITE_____ CREW SUPERVISOR_____ DATE_____

ITEM	PRESAMPLING				ON-SITE MEASUREMENT			POSTSAMPLING		
	Visual Check for Damage	Performance and/or Calibration Check	Leak Check Sampling Train	Packed for Shipment	Unpacked or Purchased On-Site	Assembled and Performance Checked	Disassembled and Packed for Shipment	Inspect for Damage and/or Excess Wear	Accepted for Future Use	To Be Replaced
SAMPLE COLLECTION										
1. Sampling Probe						+ Leak Check Sampling Train +				
2. Midget Impingers (2 ea)										
3. Drying Tube										
4. Metering Valve										
5. Vacuum Pump										
6. Dry Gas Meter										
7. Rotameter										
8. U-Connector (1 ea)										
9. Glass Elbows (2 to 4 ea)										
10. Ball Joint Clamps (4 to 8 ea)										
11. Ice Bath Container										
12. Particulate Filter (Borosilicate Glass)										
13. Ice										
14. Distilled Water										
15. Graduated Cylinder										
STACK GAS VELOCITY MEASUREMENTS										
16. Pitot Tube										
17. Diff. Pres. Gage										
18. Connecting Lines										
DOCUMENTATION										
19. Data Sheets										
BAROMETRIC PRESSURE MEASUREMENT										
20. Barometer										
CALCULATIONS										
21. Calculator/Slide Rule										
DATA VALIDATION										
22. Combustion Nomographs										

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satisfactorily completed, connect the outlet side of a calibrated wet test meter to the probe inlet forming a leakless connection. Following the same procedure as used in calibrating the dry gas meter (see subsection 2.2.2.1), make runs at rotameter settings of about 0.065, 0.075, and 0.085 ft^3/min. Calculate γ for each run (see equation in subsection 2.2.2.1 (4)). If γ at either one of the three points falls outside the range of 1.0 $\pm$ 0.02, the dry gas meter should be adjusted and recalibrated, recalibrated and a calibration curve constructed, or replaced. Record the results in the calibration log book. Date and sign the entry.

2.2.1.3 Rotameter Calibration Check. With the pump running, adjust the flow rate using the needle valve to 0.075 ft^3/min as indicated by the rotameter (use rotameter calibration curve if necessary). With a stopwatch, determine the volume registered by the dry gas meter over a time period of 2 to 5 minutes. Multiply the average flow rate indicated by the rotameter by the elapsed time in minutes and compare with the dry gas meter, volume. If the rotameter agrees within $\pm$ 5 percent of the dry gas meter, accept the rotameter calibration; otherwise, disassemble, clean, and calibrate the rotameter. Record the results in the calibration log book. Date and sign the entry.

2.2.1.4 Needle Valve Check. The needle valve should be disassembled and cleaned or replaced at signs of erratic flow-rate behavior attributable to the needle valve as observed during the above checks or when unable to regulate the flow rate at desired levels. Document the adequacy of the needle valve with a check mark in table 1 in the performance check column of the presampling phase.

2.2.1.5 Probe Heater Check. Connect the probe heating system. The probe should become uniformly hot to the touch within a few minutes after being turned on. If it does not heat properly, repair or replace as necessary. Document as part of the sampling probe performance check in table 1 for the presampling phase.

2.2.1.6 Barometer. The field barometer should be checked against a mercury barometer before each field test. If the two differ by more than $\pm$ 0.2 inches of Hg, adjust, calibrate, or replace the field barometer as applicable. Record the results in the calibration log book. Date and sign the entry.

2.2.1.7 Pitot Tube and Differential Pressure Gage. Check the velocity measuring system according to the directions given in the Quality Assurance Document for Method 2. Visual and performance checks are documented in table 1 under visual check for damage and performance and/or calibration check for the presampling phase of the field test. If a calibration check is made, it should be recorded, dated, and signed in the calibration log book.

2.2.2 Apparatus Calibration

2.2.2.1 Dry Gas Meter. The dry gas meter should be calibrated when first purchased and any time the pretest three-point check has one or more values of γ outside the range of 1.0 ± 0.02. A calibrated wet test meter, spirometer, or other standard device can be used to calibrate the dry gas meter. A wet test meter is frequently used as a laboratory standard and will be used as an example here. A 0.1 ft^3/revolution wet test meter with $\pm$ 1 percent accuracy is suitable for this calibration.

The dry gas meter can be calibrated in the following manner.

1. Assemble the apparatus as shown in figure 4-1 of appendix A with the wet test meter replacing the probe and impingers; i.e., the outlet of the wet test meter is connected to the inlet side of the silica gel tube with the inlet side of the meter vented to the atmosphere.
2. Run the pump for 15 minutes with the flow rate set at about 0.05 ft^3/min to allow the pump to warm up and to permit the interior surface of the wet test meter to be wetted.
3. Collect the information required in the form provided (fig. 2). (Sample volumes equivalent to at least one revolution of the wet test meter.)
4. Calculate γ for each flow rate setting using equation (1) and record the values in the form.

$$\gamma = \frac{V_m \left(P_m + \frac{D_m}{13.6}\right)\left(t_d + 460\right)}{V_d P_m \left(t_w + 460\right)}, \qquad (1)$$

If D_m is less than 1 inch of water, as it usually is, use the simplified relationship

DATE_____ CALIBRATED BY_____

BAROMETER PRESSURE, P_m =_____in. of Hg DRY GAS METER NO._____

WET TEST METER NO._____

Pressure Drop On W.T.M. D_m (in. of H_2O)	Rotameter Setting (ft^3/min)	Gas Vol. W.T.M. V_m (ft^3)	Gas Vol. D.G.M. V_d (ft^3)	W.T.M. t_w (°F)	D.G.M. Inlet t_{d_i} (°F)	D.G.M. Outlet t_{d_o} (°F)	D.G.M. Average t_d (°F)	Time θ (min)	γ
	0.05								
	0.06								
	0.07								
	0.08								
	0.09								
	0.10								

Figure 2. Sample form for dry gas meter calibration data.

$$\gamma = \frac{V_m (t_d + 460)}{V_d (t_w + 460)}$$

where γ = The ratio of volumes measured by the wet test meter and the dry gas meter, dimensionless,

V_m = Volume measured by wet test meter, ft^3,

P_m = Barometric pressure at the meters, inches of Hg,

D_m = Pressure drop across the wet test meter, inches of H_2O,

t_w = Temperature of wet test meter, °F,

V_d = Volume measured by the dry gas meter, ft^3, and

t_d = Average temperature of dry gas meter, °F.

5. The dry gas meter should be adjusted and recalibrated if one or more values of γ fall outside the interval 1.0 ± 0.02. Otherwise, accept the calibration as good, and forward the completed form to the supervisor for approval, then file it in the calibration log book.

2.2.2.2 Rotameter Calibration. The rotameter indication is used to set an

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approximate flow rate to start the sample collection and to maintain a sampling flow rate proportional to the stack gas velocity during sample collection.

The rotameter should be calibrated while in the sampling train as shown in figure 4-1 of appendix A. Adjust the flow rate until the rotameter indicates 90 percent of full scale. Determine the volume (ft^3) registered by the dry gas meter in 1 minute (this then is equal to the flow rate in ft^3/min). Repeat the procedure for rotameter readings of 70, 50, and 30 percent of full scale. Plot a calibration curve of rotameter reading versus flow rate. Use this calibration curve until the one-point pretest check (subsec. 2.2.1.3) differs by more than $\pm$ 5 percent from the curve. Date and sign the calibration curve and file it in the calibration log book.

2.2.2.3 Type-S Pitot Tube and Differential Pressure Gage. When used for this method alone, it is not necessary to calibrate the pitot tube unless it is desired to sample isokinetically. In the event that the pitot tube will be used for a velocity traverse or for isokinetic sampling, it should be calibrated according to the Quality Assurance Document for Method 2. In any case, the type-S pitot tube should be calibrated in the same configuration that it will be used in the field; i.e., strapped to the sampling probe while sampling at the average flow rate normally used in the field. Preliminary data indicate that if a minimum separation between the tube and probe tips of 1/2 inch or more is maintained during sampling, the influence of the sampling probe is minimized and the pitot tube can be calibrated separately; i.e., without the sampling probe. Also, if the calibration coefficient, C_p, of the type-S pitot tube falls outside the interval of 0.85 + 0.02 and is used for isokinetic sampling, the correction factor obtained from the nomograph must be multiplied by $(C_p/0.85)^2$ before determining ΔH.

2.2.3 Package Equipment for Shipment

This aspect of the test method in terms of logistics, time of sampling, and quality of data is very dependent upon the packing of the equipment in regards to 1) accessibility in the field, 2) ease of movement on site, and 3) optimum functioning of measurement devices in the field. Equipment should be packed under the assumption that it will receive severe treatment during shipment and field operation. Each item can be packaged as follows:

1. Probe, pump, dry gas meter, pitot tube, and differential pressure gage should be packed in individual cases or wooden boxes filled with packing material or lined with styrofoam.
2. Rotameter, needle valves, and all glass parts should be individually packed surrounded with suitable packing material in a shipping container.
3. Miscellaneous material, such as borosilicate (glass wool) filter to serve as a particulate filter, data sheets, and nomographs should be packed in suitable boxes or containers.

All boxes, crates, and containers should be labeled with all contents listed for easy identification.

2.3 ON-SITE MEASUREMENTS

The on-site measurement activities include transporting the equipment to the test site, unpacking and assembling the equipment, making the moisture determination, and inspecting and repacking the equipment for shipment back to the home laboratory.

2.3.1 Transport of Equipment to the Sampling Site

The most efficient means of transporting or moving the equipment from floor level to the sampling site as decided during the preliminary site visit should be used to place the equipment on-site. Care should be exercised against damage to the test equipment during the moving phase.

2.3.2 Assembly of the Test Equipment

Unpack the equipment and visually check for signs of damage sustained during shipment or transporting.

2.3.2.1 Moisture-sampling Train. Assemble the sampling train as depicted in figure 4-1 of appendix A. Leak-check the train in the same manner as was done in subsection 2.2.1.1. Also, check the probe heating system. The probe should be hot to the touch over its entire length after it has been turned on for a few minutes.

2.3.2.2 Barometer. Set up the barometer and check for proper operation by calling the nearest airport or weather bureau for the station pressure. Accept the barometer reading if they agree within $\pm$ 0.6 inches of Hg. Pressure as reported by airports and weather bureaus is usually corrected

to sea level; the uncorrected or station pressure must be requested for this use. This assumes that the sampling site and weather station are at approximately the same elevation.

2.3.3 Sample Collection

Directions are given for proportional sampling. If the stack gas velocity is relatively constant, a constant sampling rate must be maintained. Fill in the test identification data called for on the form in figure 3.

A step by step procedure is as follows:

1. Attach a type-S pitot tube to the sampling probe. Connect the pitot tube to an inclined manometer.
2. Perform a preliminary velocity traverse of the stack to get an estimate of the maximum and minimum values of ΔP to be expected.
3. Take the square root of the maximum ΔP and assign a rotameter setting (flow rate) to this value of $(\Delta P)^{1/2}$. A value of 0.090 is used here as an example. (A value no larger than about 0.085 or 0.090 ft^3/min should be used so as not to go off scale if higher ΔP's are encountered.)
4. During the test the needle valve is adjusted to provide a flow rate (Q) roughly equal to

$$Q = Q_{max} \left[\frac{\Delta P_i}{\Delta P_{max}} \right]^{1/2}$$

where Q is the flow rate read from the rotameter, $(\Delta P_i)^{1/2}$ is the square root of the instantaneous velocity pressure read from the inclined manometer, and Q_{max} and $(\Delta P_{max})^{1/2}$ are the values decided in (3) above.

5. Measure out exactly 10 ml (V_i) of distilled water in the 25-ml graduated cylinder. By estimate, transfer 5 ml to each of the impingers. Record V_i on the form in figure 3. Reconnect the impingers in the sampling train making certain that the connections are leak free.
6. Place a plug of glass wool (borosilicate filtering fiber) in the probe tip to act as a particulate filter. Use crushed ice and water to prepare an ice bath for the impingers.

TEST IDENTIFICATION

PLANT: NAME _____ LOCATION _____

STACK NO. _____ DATE OF TEST _____

TEAM: LEADER _____ OPERATORS _____, _____

RECORDED TEST DATA

CLOCK TIME	D.G.M. READING (ft^3)	VELOCITY HEAD ΔP(in. of H_2O)	$(\Delta P)^{1/2}$	ROTAMETER SETTING (ft^3/min)	METER TEMPERATURE (°F + 460)
VOLUME (V_m) _____				AVERAGE: T_m _____	

BAROMETRIC PRESSURE P_m _____ in. of Hg

MEASURES RESULTS

1. V_i _____ ml, V_f _____ ml
2. $V_{wc} = 0.0474\ ft^3/ml\ (V_f - V_i)$ = _____ ft^3
3. $V_{mc} = 17.71 \frac{°R}{in.of\ Hg} \frac{V_m P_m}{T_m}$ = _____ ft^3(dry at standard conditions)
4. $B_{wo} = \frac{V_{wc}}{V_{wc} + V_{mc}} + (0.025)$ = _____ (dimensionless)

COMPARISON DATA

1. B_{wo} _____(Calculated from combustion nomographs)
2. B_{wo} _____(For saturation at stack temperature and pressure)

Figure 3. Sample data form for moisture determination.

7. Place the sampling probe in the sampling port with the probe tip at least 12 inches from the stack wall or at the center of the stack if less than 24 inches in diameter. Plug the sampling port as well as possible with a rag, sponge, etc.
8. Sample at a rate of 0.075 ft^3/min or, if the stack gas velocity varies, at a rate proportional to the stack gas velocity according to the relationship of (4) above.
9. In proportional sampling, ΔP should be read at least every 5 minutes and appropriate flow rate adjustments made if necessary. (Adjustment in Q should be made any time the velocity pressure changes by $\pm$ 10 percent of its previous value.)
10. Continue sampling until the dry test meter registers 1 ft^3 or until visible droplets are carried over from the first impinger to the second.
11. Visually check the exit end of the sampling probe for condensation frequently during the test. Raise the probe temperature if condensation occurs during the test. If a small amount of condensate is present in the probe at the conclusion of a test, drain it into the impingers. The ice pack should be checked periodically and more ice added, if needed, during the test.
12. Record all test data on the form in figure 3.

    Note: It is recommended that the data be recorded in duplicate and that one copy be mailed to the home laboratory and that the other copy be hand carried.

2.3.4 Analysis

If the moisture content determination is to be used only for calculating the stack gas molecular weight on a wet basis, it is sufficient to quantitatively transfer the contents of both impingers to the graduated cylinder and read the volume (V_f) to the nearest 0.5 ml as directed in the reference method.

When the moisture content determination is to be used for isokinetic sampling, it is recommended that either 1) a volumetric device capable of being read to the nearest 0.1 ml be used for measuring V_i and V_f, or 2) the volume of collected water be determined gravimetrically using a balance with an accuracy of $\pm$ 0.1 g.

The volume of water vapor collected in cubic feet at standard conditions would be determined gravimetrically by

$$V_{wc} = 0.0474 \text{ ft}^3/\text{g } (W_f - W_i)$$

where V_{wc} = Volume of water vapor collected (standard conditions), ft^3

W_i = Combined weight to the nearest 0.1 g of the two impingers and the initial volume of water, g

W_f = Combined weight to the nearest 0.1 g of the two impingers and final volume of water, g.

Record V_i and V_f and method of analysis on the form in figure 3.

2.3.5 Calculations

Since moisture content is required in subsequent sampling, it is necessary to perform the calculations on-site.

Perform the calculations as indicated under measured results on the form in figure 2. All measured values (i.e., V_{wc}, V_{mc}, and B_{wo}) as well as V_m should be rounded to three significant digits and recorded in figure 3, or similiar form.

2.3.6 Data Validation

Missing data will be detected and thus obtained, if possible, while performing the above calculations. In addition, the results can be validated or at least checked for reasonableness by:

1. Comparing the measured value for B_{wo} with that value calculated using combustion nomographs and process data. The validity of the comparison depends on the accuracy of the process data. Agreement should generally be better than $\pm$ 2 percent (absolute) between the measured and calculated values of B_{wo}. If the difference is as great as 4 percent absolute, and there is confidence in the process data, it would be advisable to repeat the moisture determination. Record the measured and calculated values of B_{wo} on the form in figure 3. Combustion nomographs are available commercially for this purpose (ref. 1).

2. If the measured value appears too high from the above comparison, can be further checked by comparing it with the moisture content for saturation at stack temperature and pressure. This saturation value of B_{wo} should be recorded on the form in figure 3. A new determination should be made if the measured value exceeds or perhaps even approaches the saturation value.

2.3.7 Inspect and Pack Equipment for Return to Laboratory

As the equipment is disassembled, visually inspect each item for signs of damage and/or malfunction that were not detected during the test. If a piece of equipment was unknowingly damaged during the test, it should be documented and, if applicable, calibrated or replaced upon arrival at the laboratory. Fill in table 1 as each item is inspected and packed.

All equipment should be repacked in original containers for shipment to the laboratory.

2.4 POSTSAMPLING OPERATION

One copy of the form in figure 3 approved and signed by the supervisor should be filed in the laboratory log book and another copy forwarded for further internal review or to the user.

SECTION III MANUAL FOR FIELD TEAM SUPERVISOR

3.0 GENERAL

The term "supervisor" as used in this document applies to the individual in charge of a field team. He is directly responsible for the validity and the quality of the field data collected by his team. He may be a member of an organization that performs source sampling under contract to government or industry, a government agency performing source sampling, or an industry performing its own source sampling activities.

It is the responsibility of the supervisor to identify sources of uncertainty or error in the measuremen process for specific situations and, if possible, to eliminate or minir ze them by applying appropriate quality control procedures to insure that the data collected are of acceptable quality. These guidelines cannot cover all possible situations; therefore, it is important for the supervisor to make full use of his experience and knowledge to insure the collection of data of acceptable quality. Specific actions and operations required of the supervisor for a viable quality assurance program include, but are not limited to, the following:

1. Monitor/Control Data Quality
   a) Direct the field team in performing field tests according to the procedures given in the Operations Manual.
   b) Perform or qualify results of the quality control checks (i.e., insure that checks are valid).
   c) Perform necessary calculations and compare quality control checks with suggested performance criteria.
   d) Make corrections or alter operations when suggested performance criteria are exceeded.
   e) Forward qualified data for additional internal review or to user.
2. Routine Operations
   a) Obtain from team members immediate reports of suspicious data or malfunctions. Initiate corrective action or, if necessary, specify special checks to determine the trouble; then take corrective action. Document corrective action taken.

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b) Examine the team's log books periodically for completeness and adherence to operating procedures.

c) Approve data sheets, calibration sheets, etc., for filing.

3. Evaluation of Operations

a) Evaluate available alternative(s) for accomplishing a given objective in light of experience and needs.

b) Evaluate operator training/instructional needs for specific operations.

Consistent with the realization of the objectives of a quality assurance program as given in section I, this section provides the supervisor with brief guidelines and directions for:

1. Collection of information necessary for assessing data quality on an intrateam basis;
2. Isolation, evaluation, and monitoring of major components of system error;
3. Collection and analysis of information necessary for controlling data quality.

3.1 ASSESSMENT OF DATA QUALITY

The supervisor has at his disposal several checks that he can perform to validate data and to estimate an upper bound of the precision of the measurement process. The approach taken here was to perform a functional analysis of the measurement process (subsec. 4.1) to arrive at a precision estimate, expressed as a standard deviation, that would bracket the precision characteristic of qualified and conscientious field teams. Precision as used here then is a measure of the reproducibility of the measurement process. Once the precision estimate was arrived at and the important parameters were identified, recommended operating procedures were written (sec. II) and equipment performance criteria were recommended (table 2). For field tests in which the operating procedures are adhered to and performance criteria are satisfied, the precision is assumed to be as good as that derived from the functional analysis.

Note that the above statement deals only with precision. The measurements could be biased. As a check against large biases, it is recommended that the supervisor or crew member, through previous experience and/or knowledge of the source being tested, estimate what the moisture content

should be and compare it with the measured value. Three checks are discussed; the supervisor should select the one(s) appropriate for a particular situation. The three checks are:

1. Calculate a theoretical moisture content using process information and combustion nomographs. Combustion nomographs and instructions for making moisture content calculations are available commercially (ref. 1). In most situations, it is estimated that the theoretical value should be within $\pm$ 2 percent (absolute) of the true value.

2. Check by measuring with a different method. The wet bulb-dry bulb method is applicable for measuring moisture content of stack gases from many sources. Smith and Grove (ref. 1) discuss the usefulness and limitations of this method and present a psychrometric nomograph which allows for pressure corrections.

3. If the measured moisture content is suspected of being too high, it should be compared with the saturation value at stack temperature and pressure. The measured value should never be greater than saturation at stack conditions.

Other checks may be more appropriate in certain situations; if so, the supervisor should use that check. All checks should be documented as to type of check and the results obtained and forwarded with the data sheet.

Also it should be remembered that according to the functional analysis of subsection 4.1, an error of 1 percent (absolute) in B_{wo} results in an error of about 1 percent (relative) in isokinetic sampling, V_n/V_S, and much less than 1 percent (relative) in the molecular weight on a wet basis, M_S. Therefore, although the relative error in B_{wo} for small values of B_{wo} is large, e.g., 13 percent (relative) at B_{wo} = 11 percent (absolute), the resulting relative effect on V_n/V_S and M_S is much smaller.

3.1.1 Required Information

Only the supervisor can judge if the recommended operating procedures have been deviated from in such a manner and to such a degree that the precision statement as proposed here is invalid. If unavoidable and/or uncontrolled deviations occur, they should be documented and forwarded with the data sheet and no precision statement made. An estimate of the upper bounds for error should be made and forwarded with the data when possible.

Results of the calibration checks for the dry gas meter, barometer, and thermometer made prior to the field test are compared with the performance criteria of table 2 to verify that the criteria have been satisfied.

3.1.2 Reporting Data Quality

For field tests in which recommended operating procedures were followed, equipment performance criteria were satisfied, and special quality checks did not indicate that gross errors were present, the standard deviation derived from the functional analysis is taken as a realistic upper bound for the precision of the field data. An average standard deviation of 1.2 percent (absolute) was obtained over a moisture content range from about 10 to 35 percent (see subsec. 4.1.1). Reporting the measured moisture content, B_{wo}, with $\pm$ 3 σ limits then would be

$$B_{wo} \pm 3.6 \text{ percent (absolute)}.$$

The utility of the above statement follows from the fact that if the measured values of B_{wo} are normally distributed about a true value B_{wo_t} (assuming no bias) with $\sigma\{B_{wo}\} = 1.2$ percent (absolute), then there is approximately 99.7* percent confidence that B_{wo_t} would be in the above interval. This statement is about precision only and is based on a measure of reproducibility of measurements of B_{wo} for teams adhering to the quality assurance guidelines of this document.

3.2 SUGGESTED PERFORMANCE CRITERIA

Data assessment as discussed in the previous subsection was based on the premise that all variables were controlled at a given level and that good operating practices were followed. These levels of suggested performance criteria are the values given in the Operations Manual for determining when equipment variability is excessive and needs correcting. Criteria for judging performance are summarized in table 2.

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*Throughout this report the normal distribution is used to obtain confidence limits for the mean whenever the standard deviation is based on assumed values; e.g., the value derived from the variance analysis (subsec. 4.1.1). However, if the standard deviation is estimated from a finite sample, the t-distribution should be used to obtain confidence limits.

Table 2. Suggested performance criteria

1. Suggested Criteria for Equipment Performance
   a) Dry test meter: $\gamma = 1.0 \pm 0.02$
   b) Barometer: $\pm$ 0.2 in. of Hg
   c) Thermometer (meter): $\pm$ 5°R at 460°R
2. Suggested Criteria for Performing Calibration
   a) Dry test meter. Perform a full calibration when new, before every third field test, before any test in which the meter has not been calibrated in 3 months, at any sign of damage, or when obviously wrong results are obtained. It is highly recommended that a three-point calibration check be performed before each field test and if γ for any point is outside the 1.0 ± 0.02 limits, a total calibration be performed.
   b) Barometer. Before each field test, the barometer should be compared with a wall-mounted mercury bulb barometer and adjusted if the difference is greater than 0.2 in. of Hg.
   c) Thermometer. Check by measuring the temperature of an ice bath before each field test.

3.3 COLLECTION AND ANALYSIS OF INFORMATION TO IDENTIFY TROUBLE

In a quality assurance program, one of the most effective means of preventing trouble is to respond immediately to indications of suspicious data or equipment malfunctions. Certain visual and operational checks can be performed while the measurements are being made to help insure the collection of data of good quality. These checks are written as part of the routine operating procedures in section II. In order to effectively apply preventive-type maintenance procedures to the measurement process, the supervisor must know the important variables in the process, how to monitor them, and how to interpret the data obtained from monitoring operations. These subjects are discussed in the following subsections.

3.3.1 Identification of Important Variables

Determination of moisture content in stack gases requires a sequence of operations and measurements that yields as an end result a number that serves to represent the average moisture content. There is no way of

knowing the accuracy, i.e., the agreement between the measured and the true value, for a given field test. However, a knowledge of the important variables and their characteristics allows for the application of quality control procedures to control the effect of each variable at a given level during the field test, thus providing a certain degree of confidence in the validity and accuracy of the final result.

A functional analysis of this method of measuring the moisture content of stack gases was made to try to identify important components of system error. Individual error components are estimated using engineering judgment in a manner such that their combined variability is consistent with overall system error.

Three operational-type errors judged to be important are: 1) volumetric (water) reading error; 2) failure to quantitatively transfer water from the graduated cylinder to the impingers and vice versa; and 3) in cases where the silica gel tube is not used or is not weighed before and after sample collection, an error could result from failure to maintain a condenser temperature of 70°F or less; i.e., the gas temperature as it leaves the last impinger. These errors would be expected to be of a random nature and to increase in magnitude as field conditions become more adverse.

Other sources of error include: 1) failure to collect a representative sample; e.g., inability to maintain a proportional sampling rate; 2) leaks in the sampling train; 3) dry gas meter inaccuracy; and 4) moisture condensing out in the (heated) probe before reaching the impinger. These errors are or should be minimized by adhering to good operating practices as set forth in the Operations Manual.

A brief description of the assumptions made and the techniques used in the functional analysis is given in subsection 4.1 of this document. A more comprehensive treatment of combining error terms will be given in the final report for this contract. The source and magnitude of uncertainty for each of the above parameters are discussed below.

3.3.1.1 Volumetric (Water) Errors. Volumes are read to the nearest 0.5 ml. This means that in some cases the volume error due to rounding will be as much as 0.25 ml. Under typical field conditions a reading error distribution characterized by a standard deviation of 0.25 ml is assumed. For small volumes of collected water, e.g., if $B_{wo} = 0.20$ and 1 ft^3 of stack gas is sampled, a total of about 4 ml of water would be collected in the

impingers. The relative standard deviation then would be 6 percent (i.e., 0.25 × 100/4 = 6.25). Because of the small volume involved, it is recommended that graduated cylinders with divisions of 0.2 ml be used or that the water volume be determined gravimetrically, especially when the data are to be used in isokinetic sampling.

3.3.1.2 Condenser Temperature. The reference method assumes a gas temperature of about 70°F as it exits from the second impinger and allows for the proportion of water vapor in air at standard pressure. In situations where the gas temperature is not controlled at 70°F and/or the exit pressure is significantly different from atmospheric pressure, the constant correction factor will be wrong. Again, for a true $B_{wo} = 0.20$ for the stack gas, an exit temperature of 90°F at standard pressure has a volumetric proportion of water vapor at saturation of 0.048. Using 0.025 would result in an 11 percent error in B_{wo}. An error in the opposite direction results when the exit temperature is less than 70°F. There is no easy way of monitoring the exit gas temperature. Maintaining an ample quantity of ice water in the ice bath container and observing the drying tube for the degree and quantity of color change of the silica gel are two means of insuring that the temperature does not become too high. The exit gas temperature varied from 50° to 90°F for four sampling trains and six runs each for Method 5 (ref. 1). The residence time of the gas in the impingers and ice bath is longer for Method 5 than for the sampling train of Method 4; therefore, it is reasonable to assume a similar or greater temperature variation for Method 4.

3.3.1.3 Proportional Sample. If the stack gas velocity varies with time, the sampling rate must be maintained proportionally. Failure to comply with this requirement will result in a nonrepresentative sample. In proportional sampling, the velocity pressure head should be read and recorded at least every 5 minutes and flow-rate adjustments should be made any time $(\Delta P)^{1/2}$ changes significantly. The deviation of the measured value from the true value depends on the magnitude and rapidity of change of the stack gas velocity and the degree to which the crew can maintain proportional flow rates.

This error is not estimated here. A check on how well the operator maintained proportional sampling throughout the test can be made if proper

records are maintained during sampling; i.e., the rotameter reading, Q, divided by the square root of the velocity pressure, $(\Delta P_i)^{1/2}$, should be a constant throughout the test.

3.3.1.4 Sampling Train Leaks. The sampling train is assembled and leak-checked before water is placed in the impingers. Care must be exercised in remaking the two connections after water has been added to insure a leak-free train. If it is desired to leak-check the train with water in the impingers, no more than 15 inches of Hg vacuum should be drawn or the water will boil and saturate the silica gel. This is a relatively simple train, and, with proper care, leaks should be negligible, certainly less than 1 percent of the average flow rate.

3.3.1.5 Dry Gas Meter Accuracy. A dry gas meter, when properly maintained and calibrated, is reported to have an accuracy of $\pm$ 2 percent with a precision of less than 1 percent. Operating under field conditions, a relative standard deviation of 1 percent seems reasonable for the precision of the volume as measured by the dry gas meter.

3.3.2 How to Monitor Important Variables

In general, if the procedures outlined in the Operations Manual are followed, the major sources of systematic error contributing to measurement bias will be in control. It is felt, however, that the supervisor should visually check certain parameters and operations periodically while measurements are being made to insure the use of proper equipment and technique.

Five subjects are considered important enough to be specially checked by the supervisor at least once during each test: 1) proportional sampling, 2) collected water volume, 3) probe temperature, 4) condenser temperature, and 5) dry gas meter readings.

3.3.2.1 Proportional Sampling. How well proportional sampling is being accomplished can be qualitatively evaluated by observing the magnitude of change in the velocity pressure head between successive readings. The larger the change between adjustments, the larger the error due to non-proportional sampling. It is recommended that readings and subsequent adjustments be made at least every 5 minutes. However, due to the time required to make the necessary readings, calculations, adjustments, and documentation, a time interval of less than about 1 minute probably should not be attempted regardless of the variability in the velocity pressure

head. Also, check to see that the flow rate is being adjusted relative to the change in $(\Delta P)^{1/2}$ and not ΔP; i.e., the ratio $Q/(\Delta P)^{1/2}$ should remain constant throughout the test.

3.3.2.2 Collected Water Volume Determination. Only two measurements are involved per test. The supervisor should check to see that an analytical device (volumetric or gravimetric) with acceptable precision and accuracy is available and is used for the volume determinations.

3.3.2.3 Probe Temperature. The probe should be visually checked at least once during sample collection for signs of condensation prior to the first impinger. The presence of condensed moisture requires that the probe temperature be increased. If condensation is present in the probe at the end of a test, the probe temperature should be increased and the test continued until the condensate is gone; if this is not feasible, the test should be repeated.

3.3.2.4 Condenser Temperature. The ice bath should be checked for an adequate quantity of ice; a slurry should be maintained. When sampling hot gases (above 250°F) and in hot weather, the ice water should cover at least three-fourths of the impinger. Salt can be added to the bath to lower the temperature further if it is suspected that the sample gas is not being cooled to 70°F. The silica gel tube should be observed for indication of excess moisture. Estimates of the quantity of silica gel affected and the degree of color change for a normal test can be made from experience. Any noticeable departure indicates that the sample gas is not saturated at 70°F as it reaches the silica gel tube.

3.3.2.5 Dry Gas Meter Readings. The volume of dry gas sampled is determined from the initial and final dry gas meter readings. One of these readings should be checked by a second crew member. Differences greater than normal reading errors, e.g., one division of the smallest scale on the meter, should be corrected; if uncorrectable, the test should be repeated.

SECTION IV MANUAL FOR MANAGER OF GROUPS OF FIELD TEAMS

4.0 GENERAL

The guidelines for managing quality assurance programs for use with Test Method 4, Determination of Moisture in Stack Gases, are given in this part of the field document. This information is written for the manager of several emission source measuring teams and for the appropriate EPA, State, or Federal administrators of these programs. It is emphasized that if the analyst carefully adheres to the operational procedures and checks of section II, then the errors and/or variations in the measured values should be consistent with the results of the functional analysis. Consequently, the auditing routines given in this section provide a means of determining whether the stack sampling test teams of several organizations, agencies, or companies are following the suggested procedures. The audit function as recommended for this method is primarily one of independently obtaining measurements and performing calculations where this can be done. The purposes of these guidelines are to:

1. Present information relative to the test method (a functional analysis) to identify the important operations and factors,
2. Present a data quality audit procedure for use in checking adherence to test methods and validating that performance criteria are being satisfied, and
3. Present the statistical properties of the auditing procedure in order that the appropriate plan of action may be selected to yield an acceptable level of risk to be associated with the reported results.

These three purposes will be discussed in the order stated in the sections that follow. The first section will contain a functional analysis of the test method with the objective of identifying the most important factors that affect the quality of the reported data and of estimating the expected variation and biases in the measurements resulting from equipment and operator errors.

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There are no absolute standards with which to compare the routinely derived measurements. Furthermore, the taking of completely independent measurements at the same time that the routine data are being collected (e.g., by introducing two sampling probes into the stack and collecting two samples simultaneously) is not considered practical due to the constrained environmental and space conditions under which the data are being collected. Hence, a combination of an on-site system audit, including visual observation of adherence to operating procedures, and a quantitative performance quality audit check is recommended as a dual means of independently checking on the source emissions data.

The second section contains a description of a data quality audit procedure. The most important variables identified in section 4.1 are considered in the audit. The procedure involves the random sampling of n stacks from a lot size of N = 20 stacks (or from the stacks to be tested during a 3-month period, if less than 20) for which one firm is conducting the source emissions tests. For each of the stacks selected, independent measurements will be made of the indicated variables. These measurements will be used in conjunction with the routinely collected data to estimate the quality of the data being collected by the field teams.

The data quality audit procedure is an independent check of data collection and analysis techniques with respect to the important variables. It provides a means of assessing data collected by several teams and/or firms with the potential of identifying biases/excessive variation in the data collection procedures. A quality audit should not only provide an independent quality check, but should also identify the weak points in the measurement process. Thus the auditor, an individual chosen for his background knowledge of the measurement process, will be able to guide field teams in using improved techniques. In addition, the auditor is in a position to identify procedures employed by some field teams which are improvements over the current suggested ones, either in terms of data quality and/or time and cost of performance. The auditor's role will thus be one of aiding the quality control function for all field teams for which he is responsible, utilizing the cross-fertilization of good measurement techniques to improve the quality of the collected and reported data. A summary of the quality audit procedures including a sample calculation is given as appendix E.

The statistical sampling and test procedure recommended is sampling by variables. This procedure is described in section 4.3. It makes maximum use of the data collected, and it is particularly adaptable to the small lot size and consequently the small sample size applications. The same sampling plans can be employed in the quality checks performed by a team or firm in its own operations. The objectives of the sampling and test procedure are to characterize data quality for the user and to identify potential sources of trouble in the data collection process for the purpose of correcting the deficiencies in data quality.

4.1 FUNCTIONAL ANALYSIS OF TEST METHOD

Test Method 4, Determination of Moisture in Stack Gases, is described in the Federal Register of December 23, 1971, and is reproduced as appendix A of this document. Under standards of performance for new stationary sources, Method 4 is used to determine the moisture content of nitric acid plant emissions for subsequent use in calculating the stack gas molecular weight on a wet basis. It can also be used to determine the moisture content for use in isokinetic sampling. The functional analysis is performed in an effort to determine the variability to expect in the determination of moisture content and its subsequent influence on setting isokinetic sampling conditions and determining the stack gas molecular weight on a wet basis. The functional analysis starts with the basic relationship of B_{wo} to the measured values obtained from the test as given by equation (2) and results in an expression for the variance B_{wo} as a function of the variances and mean values of the measured quantities given by equation (12). Also, the subsequent influence of the variability of B_{wo} on the determination of stack gas molecular weight and isokinetic sampling conditions is given by equations (14) and (15), respectively. Results of the functional analysis indicate that the molecular weight is relatively insensitive to the normally expected variability in moisture measurements. Normal variability in moisture determinations would not cause biases larger than about $\pm$ 4 percent (these are 3 CV values) in isokinetic sampling.

In Method 4 the sampling train is assembled as shown in figure 4-1 of appendix A.

The moisture content is calculated by

$$B_{wo} = \frac{V_{wc}}{V_{wc} + V_{mc}} + 0.025, \qquad (2)$$

where B_{wo} = Proportion by volume of water vapor in the gas stream, dimensionless

V_{wc} = The volume of water vapor collected at standard conditions, ft^3

V_{mc} = Dry gas volume through the meter at standard conditions, ft^3

0.025 = Volumetric proportion of water vapor at saturation in the gas stream 70°F.

The volume of water vapor collected at standard conditions, V_{wc}, is given by equation (4-1) of appendix A, namely

$$V_{wc} = 0.0474\ ft^3/ml(V_f - V_i) \qquad (3)$$

where V_f = The total volume of water in both impingers at the conclusion of the test, ml

V_i = The total volume of water in both impingers at the start of the test, ml

0.0474 = The number of cubic feet that 1 ml of water would occupy in the vapor state at standard conditions.

For this analysis it is assumed that V_i, the initial volume, is 10 ml and will be measured in the graduated cylinder, then divided equally between the two impingers. Also, the final volume, V_f, will be obtained by combining the contents of both impingers in the graduated cylinder and making one reading. The reference method specifies that V_i and V_f be read to the nearest 0.5 ml. Therefore, in some instances error due to rounding alone will approach 0.25 ml. For this analysis reading error is assumed to be a random normal deviate with a zero mean and an estimated standard deviation of 0.25 ml. This would include pure reading error as well as error due to rounding to the nearest 0.5 ml. Typical volumes of water collected, i.e., $V_f - V_i$, range from 2 to 10 ml and rounding to the nearest 0.5 ml could induce a significant error, particularly for the smaller volumes.

It is also assumed that V_i and V_f will be measured in the same graduated cylinder, thus eliminating the necessity of calibrating the cylinder and the variability between cylinders.

The dry gas volume, V_m, measured by the dry gas meter at meter conditions is corrected to the dry gas volume at standard conditions, V_{mc}, by equation (4-2) of appendix A, namely

$$V_{mc} = 17.71 \left[\frac{V_m P_m}{T_m} \right] \tag{4}$$

Moisture content as determined by equation (2) is used to calculate the stack gas molecular weight on a wet basis according to

$$M_s = M_d(1 - B_{wo}) + 18B_{wo} \tag{5}$$

where M_s = Molecular weight of the stack gas on a wet basis, lb/lb-mole

M_d = Dry molecular weight of stack gas (from Method 3), lb/lb-mole.

When used to determine isokinetic sampling rates, the moisture content is used in the relationship

$$\frac{V_n}{V_s} = \left(\frac{1}{D_n^2} \right) \left(\frac{K_m}{C_p K_p} \right) \left(\frac{1-B_{wm}}{1-B_{wo}} \right) \left[\left(\frac{P_m}{P_s} \right) \left(\frac{T_s}{T_m} \right) \left(\frac{M_s}{M_m} \right) \frac{\Delta M}{\Delta P} \right]^{1/2} \tag{6}$$

Where V_n/V_s is the ratio of the gas velosity at the sampling nozzle and the stack gas velocity. To determine the influence on V_n/V_s due to variability in B_{wo}, all terms not involving B_{wo} can be combined and treated as a constant, K'. Setting $M_s = M_d(1 - B_{wo}) + 18B_{wo}$ in equation (6) and simplifying gives

$$\frac{V_n}{V_s} = K' \frac{\left[M_d(1 - B_{wo}) + 18B_{wo} \right]^{1/2}}{1 - B_{wo}} \tag{7}$$

To further simplify, let $M_d = 30$ (M_d can only vary from about 29 to 31), then (7) becomes

$$\frac{V_n}{V_s} = K \frac{(5 - 2B_{wo})^{1/2}}{(1 - B_{wo})}, \text{ where } K = K' \sqrt{6} \qquad (8)$$

4.1.1 Variance Analysis

Using standard techniques for determining the variance of linear and nonlinear functions (ref. 2), the variances of the different measurements are estimated. The individual variances are propagated through the system mathematically to determine their combined influence on the final result.

The variance analysis is performed in a stepwise fashion, treating each parameter or variable in the model (eq. (2)) individually. If data and/or engineering judgment indicates that the variable has a constant standard deviation over its working range, then variances are determined directly such as in equation (9). However, in instances where the coefficients of variation (i.e., the relative standard deviation) of the variables are constant, the variance is determined for discrete values of the parameter of interest. An example of this is illustrated in equations (10) and (11).

4.1.1.1 Variance of Volume of Water Vapor Collected at Standard Conditions, V_{wc}. The volume of water vapor collected at standard conditions is given by equation (3). The variance of V_{wc}, assuming V_f and V_i to be independent, is given by

$$\sigma^2\{V_{wc}\} = (0.0474)^2 [\sigma^2\{V_f\} + \sigma^2\{V_i\}] \qquad (9)$$

From subsection 3.3.1.1 variances of V_f and V_i are both assumed to be $(0.25 \text{ ml})^2$, and thus the estimated variance of V_{wc} is

$$\sigma^2\{V_{wc}\} = (0.0474)^2 [(0.25)^2 + (0.25)^2] = 0.00028,$$

and the standard deviation becomes

$$\sigma\{V_{wc}\} = 0.017 \text{ ft}^3.$$

4.1.1.2 Variance of the Dry Gas Volume Through the Dry Gas Meter at Standard Conditions, V_{mc}. The variance of V_{mc} from equation (4) is obtained by first determining the coefficients of variation (sometimes referred to as the relative standard deviation) and then determining the variance and thus the standard deviation. The coefficient of variation of V_{mc} is given by

$$CV\{V_{mc}\} = [CV^2\{V_m\} + CV^2\{P_m\} + CV^2\{T_m\}]^{1/2}. \quad (10)$$

For this analysis the 3 CV value for V_m is taken as 3 percent. The performance criterion (table 2) is that it be within $\pm$ 2 percent of the wet test meter. Allowing for some inaccuracy in the wet test meter and for the various environmental conditions that the dry gas meter is subjected to in the field, a CV of 1 percent (3 CV = 3 percent) seems realistic. With the same line of reasoning, 3 CV values for P_m and T_m are taken as 3/2 times the performance criteria of table 2. The coefficients of variation then are 0.3 percent and 0.5 percent for P_m and T_m, respectively. Using these values in equation (10) gives

$$CV\{V_{mc}\} = 0.0115 = 1.15 \text{ percent (relative)}.$$

The standard deviation for discrete values of V_{mc} can be obtained from

$$\sigma\{V_{mc}\} = (CV\{V_{mc}\} \times V_{mc})/100 = 0.0115 \times V_{mc}. \quad (11)$$

For this method the volume registered by the dry gas meter (V_m) is approximately 1 ft^3. If T_m and P_m are not too different from 530°R (70°F) and 29.92 inches of Hg, respectively, then V_{mc} will also be about 1 ft^3. For this situation the standard deviation of V_{mc} at V_{mc} = 1 ft^3 is

$$\sigma\{V_{mc}\} = 0.0115 \text{ ft}^3.$$

For conditions other than those stated above, namely V_m = 1 ft^3, T_m = 530°R, and P_m = 29.92 inches of Hg, the standard deviation has to be determined by equation (11) for a given V_{mc}.

4.1.1.3 Variance of the Moisture Content, B_{wo}. The variance of B_{wo} as given by equation (2) is obtained using the following relationship:

$$\sigma^2\{B_{wo}\} = \frac{(V_{mc})^2}{(V_{wc} + V_{mc})^4}\,\sigma^2\{V_{wc}\} + \frac{(V_{wc})^2}{(V_{wc} + V_{mc})^4}\,\sigma^2\{V_{mc}\} + \sigma^2\{\varepsilon\} \qquad (12)$$

The term $\sigma^2\{\varepsilon\}$ represents the variance in the moisture content of the sample gas as it leaves the last impinger. This variation is due to temperature variations as discussed in subsection 3.3.1.2. Assuming a temperature variation from 50° to 90°F, the moisture content varies from 0.012 to 0.048, respectively. Therefore, for this analysis, $\sigma\{\varepsilon\}$ was taken as 0.005. For the set of conditions where $V_{mc} \approx 1$ ft^3, $\sigma\{V_{wc}\} = 0.017$ ft^3, $\sigma\{V_{mc}\} = 0.0115$ ft^3, and letting $\Delta V = V_f - V_i$ take on the values of 2, 6, and 10 ml, equation (12) gives $\sigma\{B_{wo}\} = 0.015$, 0.011, and 0.0097, respectively. The values of ΔV under the assumptions made above represent $B_{wo} \approx 0.112$, 0.246, and 0.347, respectively. Relative errors expressed as the coefficient of variation CV = 13, 4, and 3 percent (relative) for moisture contents of 11.2, 24.6, and 34.7 percent (absolute), respectively.

Equation (12) can be used to estimate the variance of B_{wo} for different values of V_{mc} and V_{wc} whose estimated variances are determined from equations (11) and (9), respectively.

4.1.1.4 Variance of M_s due to Variability in B_{wo}. The resulting variability in M_s as given by equation (5) from variations in B_{wo} can be ascertained from the relationship

$$\sigma^2\{M_s\} = (1 - B_{wo})^2\,\sigma^2\{M_d\} + (18 - M_d)^2\,\sigma^2\{B_{wo}\} \; . \qquad (13)$$

The estimated variance of M_d is taken from the quality assurance document of this series dealing with Method 3. That value is $\sigma^2\{M_d\} = 0.04$ (lb/lb-mole)^2. M_d is taken as 30 lb/lb-mole (about the midpoint of the possible range), B_{wo} depends on ΔV and for a value of $\Delta V = 2$ ml, $\sigma\{B_{wo}\}$ is taken as 0.015 and used in equation (13) to give

$$\sigma^2\{M_s\} = 0.789(0.2)^2 + 144(0.015)^2 = 0.0640 \qquad (14)$$

and

$$\sigma\{M_s\} = 0.253 \text{ lb/lb-mole.}$$

Taking a mean value of $\bar{M}_s = 28.7$ lb/lb-mole, the coefficient of variation is seen to be $CV\{M_s\} = (0.253/28.7) \times 100 = 0.88$ percent. Calculations showed $CV\{M_s\} < 1$ percent for B_{wo} values from about 0.10 to 0.35.

4.1.1.5 Variance of V_n/V_s due to Variation in B_{wo}. In order to estimate the effect of variation of B_{wo} on isokinetic sampling, equation (8) is used in conjunction with the variance of B_{wo} as previously obtained to yield equation (15) below relating $\sigma^2\{V_n/V_s\}$ to the corresponding data for B_{wo},

$$\sigma^2\left\{\frac{V_n}{V_s}\right\} = K^2 \frac{(4 - B_{wo})^2}{(1 - B_{wo})^4(5 - 2B_{wo})} \sigma^2\{B_{wo}\} . \qquad (15)$$

Substituting the value of 0.015 for $\sigma\{B_{wo}\}$ in (15) gives $\sigma\{V_n/V_s\} = 0.0332K$ for $B_{wo} = 0.112$, $0.346K$ for $B_{wo} = 0.246$, and $0.40K$ for $B_{wo} = 0.347$. The corresponding coefficients of variation, $CV\{V_n/V_s\}$, are 1.4, 1.2, and 1.3 percent, respectively. From these calculations and those of subsection 4.1.1.3, it can be seen that errors in B_{wo} of 1.5, 1.1, and 0.97 percent (absolute) result in relative errors in V_n/V_s of 1.4, 1.2, 1.3 percent, respectively.

4.1.1.6 Summary of the Variance Analysis. Models for determining the variance of B_{wo}, (eq. (12)) and the variances of the M_s (eq. (13)), and V_n/V_s (eq. (15)) are presented in the previous subsections. The assumed means and standard deviations/coefficients of variations used in this analysis are summarized in table 3. In the table the applicable equation number for calculating the variance is given below the variable. The variance of B_{wo} is sensitive to the volume of water collected, and the results are given for collected volumes (ΔV_s) of 2, 6, and 10 ml.

4.2 PROCEDURES FOR PERFORMING A QUALITY AUDIT

"Quality audit" as used here implies a comprehensive system of planned and periodic audits to verify compliance with all aspects of the quality assurance program. Results from the quality audit provide an independent assessment of data quality. "Independent" means that the individuals performing, as much as possible of the equipment, and maybe even the measuremen

method used in the audit are different from the regular field crew, equipment, and method. From these data, inferences can be made concerning the bias and precision of field data.

The auditor, i.e., the individual performing the audit, should have extensive background experience in source sampling, specifically with the characterization technique that he is auditing. He should be able to establish good rapport with field crews.

Table 3. Summary of variance analysis computations

Variable	Statistic	$\Delta V = V_f - V_i$, ml.		
		2 ml.	6 ml.	10 ml.
V_{wc} (ft^3)	Mean	0.095	0.190	0.284
eq. (9)	σ	0.017	0.017	0.017
	CV (%)	17.9	8.9	6.0
V_{mc} (ft^3)	Mean	*	≈ 1	*
eqs. (10) and (11)	σ		0.0115	
	CV (%)		1.15	
B_{wo} (%)	Mean	11.2	24.6	34.7
eq. (12)	σ	0.015	0.011	0.0097
	CV (%)	13	4	3
M_d (lb/lb-mole)	Mean	*	30	*
(From Method 3	σ		0.2	
Document)	CV (%)		0.67	
M_s (lb/lb-mole)	Mean	28.7	27.0	25.8
eq. (13)	σ	0.253	0.235	0.222
	CV (%)	0.88	0.87	0.86
V_n/V_s (dimensionless)	Mean	2.37 K	2.88 K	3.08 K
eq. (15)	σ	0.0332 K	0. 0346 K	0.040 K
	CV (%)	1.4	1.2	1.3

*Use same values for all levels of ΔV.

Because the normal variability in measuring B_{wo}, as derived from the functional analysis, has a relatively small influence on M_s and even less on V_s, it is felt that this method can be adequately audited by on-site observation of adherence to recommended operation procedures and comparison of test results with theoretical values and /or results obtained with wet bulb and dry bulb thermometers and psychrometric charts in conjunction with laboratory calibration records showing that equipment performance criteria have been satisfied.

A summary of the audit procedure with a sample calculation is given in appendix E.

The functions of the auditor are summarized in the following list:

1. Observe procedures and techniques of the field team during on-site measurements.
2. Check/verify applicable records of equipment calibration checks in the field team's home laboratory.
3. Perform calculations using data obtained from the audit.
4. Compare the audit value with the field team's test value.
5. Inform the field team of the comparison results specifying any area(s) that need special attention or improvement.
6. File the records and forward the comparison results with appropriate comments to the manager.

4.2.1 Frequency of Audit

The optimum frequency of audit is a function of certain costs, quality of the incoming data, and desired level of confidence in the data quality assessment. A methodology for determining the optimum frequency using relevant costs is presented in the Quality Assurance Documents of this series dealing with Pollutant Specific Methods requiring the results of Method 4 and in the final report of this contract. Costs will vary between field teams and types of field tests. Therefore, the most cost-effective auditing level will have to be derived using relevant local cost data according to the procedure given in the final report on this contract.

4.2.2 Collecting On-site Information

While on-site, the auditor should observe the field team's overall performance of the field test. Table 4 is a sample check list of the operations to observe. Each item on the list should be checked yes or

Table 4. Moisture determination checklist to be used by auditor

YES	NO	OPERATION
		PRESAMPLING PREPARATION
___	___	1. Sampling train assembled as shown in figure 4-1 of appendix A.
___	___	2. Sampling train leak checked by plugging the probe tip prior to adding water to impingers.
___	___	3. Measurement and quantitative transfer of 10 ml (about 5 ml each) to the impingers.
___	___	4. Sufficient ice and water in impinger bath.
		SAMPLE COLLECTION
___	___	5. Probe tip at stack center or no closer than 12 inches to the stack wall.
___	___	6. Sampling port adequately plugged.
___	___	7. Process at correct operating level.
___	___	8. Signs of condensation in the probe during sample collection.
___	___	9. Minimum sample volume as read by dry gas meter, 1 ft^3.
___	___	10. Excessive quantity of silica gel turned pink indicating insufficient cooling of sample gas in impingers.
___	___	11. Proportional sampling, if applicable.
		ANALYSIS
___	___	12. Contents of impingers quantitatively transferred to graduated cylinder.
___	___	13. Same graduated cylinder used for measuring V_f and V_i.
		DOCUMENTATION
___	___	14. All information recorded on data sheet as obtained.
___	___	15. Any unusual conditions recorded.
		COMMENTS

no according to whether they were performed according to the Operations Manual or not. Those checked no should be explained under Comments. No check list can cover all situations; the auditor should include other checks as deemed desirable for a specific situation.

In addition to the on-site observations of table 4, it is recommended that the auditor either measure the moisture content of the stack gas using the wet and dry bulb method or calculate a theoretical moisture content using process data or by performing a material balance. The method most appropriate to the source being tested should be used by the auditor. For the discussion to follow on comparing audit and routine results, it is assumed that the method used by the auditor has about the same variability as the reference method; i.e., approximately 68 percent of the time, the value of B_{wo} as determined by the auditor is within $\pm$ 1.2 percent (absolute) of the true value, $\pm$ 2.4 percent (absolute) 95 percent of the time, and $\pm$ 3.6 percent (absolute) about 99.7 percent of the time. (Assuming a standard deviation $\sigma\{B_{wo}\}$ = 1.2 percent (absolute)). If for a particular source the auditor feels that neither of the above methods would be as accurate as the reference method, this should be stated in his report, and the following comparison would not be made.

The audit and routine (field team's results) values are compared by

$$d_j = (B_{wo_j} - B'_{wo_j}) \times 100 \qquad (16)$$

where d_j = The difference in the audit and field test results for the jth audit, percent (absolute)

B_{wo_j} = Moisture content of the stack gas as measured by the field team, proportion by volume

B'_{wo_j} = Moisture content of the stack gas as measured and calculated by the auditor, proportion by volume.

Record the value of d_j in the quality audit log book. Also, it is recommended that the d_j's be plotted on a quality control chart as shown in figure 4. These limits assume equal variability in the audit method and the reference method. After 20 to 25 values are obtained using the same audit method, these limits can be reevaluated and tightened if possible. Quality control charts are discussed in textbooks such as references 3 and 4.

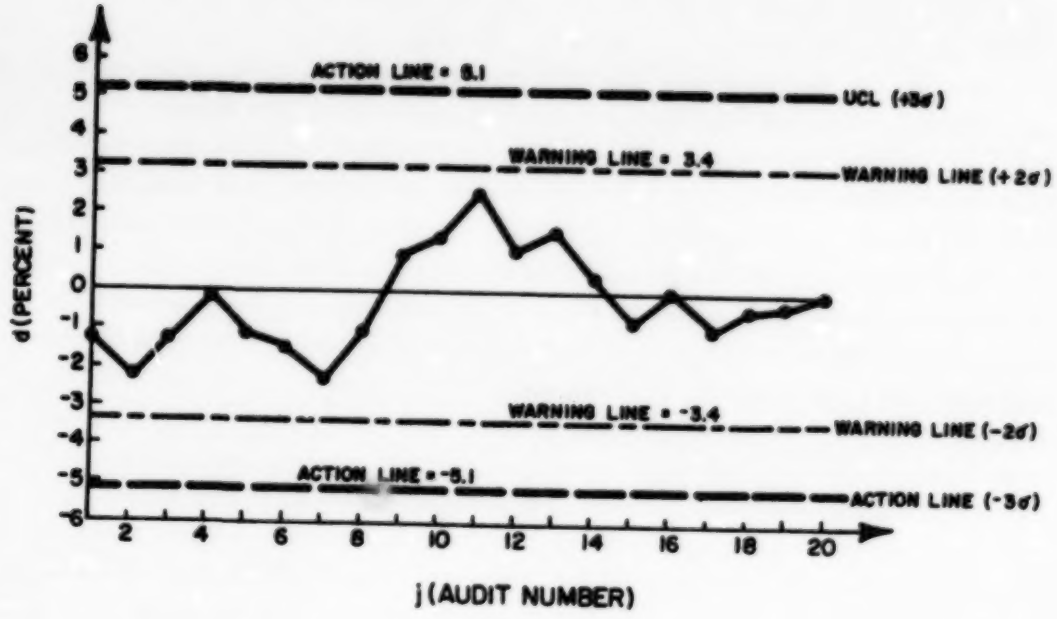

Figure 4. Sample control chart for audit data.

4.2.3 Collecting Laboratory Information

When visiting the field team's home laboratory, the auditor should verify by checking the calibration records that the performance criteria as given in table 2 of section II have been met over the period since the last audit was performed.

4.2.4 Overall Evaluation of Field Team Performance

In a summary-type statement, the field team should be evaluated on its overall performance. Reporting the d_j value as previously computed is an adequate representation of the objective information collected for the audit. However, unmeasurable errors can result from nonadherence to the prescribed operating procedures and/or from poor technique in executing the procedures. These error sources have to be estimated subjectively by the auditor. Using the check list filled out in the field (table 4), the team could be rated on a scale of 1 to 5 as follows:

5 - Excellent

4 - Above average

3 - Average

2 - Acceptable, but below average

1 - Unacceptable performance.

In conjunction with the numberical rating, the auditor should include justification for the rating. This could be in the form of a list of the team's strong/weak points.

4.3 DATA QUALITY ASSESSMENT

Two aspects of data quality assessment are considered in this section. The first considers a means of estimating the precision and accuracy of the reported data; e.g., reporting the bias, if any, and standard deviation associated with the measurements. The second consideration is that of testing the data quality against given standards using sampling by variables. For example, lower and upper limits, L and U, may be selected to include a large percentage of the measurements and outside of which it is desired to control the percentage of measurements to, say, less than 10 percent. If the data quality is not consistent with these limits, L and U, then action is taken to correct the possible deficiency before future field tests are performed and to correct the previous data when possible.

4.3.1 Estimating the Precision/Accuracy of the Reported Data

A method for estimating the precision (standard deviation) of the moisture content measurements was given in section 4.1. This section will indicate how the audit data collected in accordance with the procedure described in section 4.2 will be utilized to estimate the precision and bias of the measurements. Similar techniques can also be used by a specific firm or team to assess their own measurements. However, in this case no bias data can be obtained. The audit data collected as a result of following the procedures in the previous section are

$$d_j = (B_{wo_j} - B'_{wo_j}) \times 100 .$$

These are differences between the field team results and the audited results. Let the mean and standard deviation of the difference d_j and $j = 1, \ldots, n$ be denoted by $\bar{d}$ and s_d, respectively.

Thus

$$\bar{d} = \sum_{j=1}^{n} d_j/n \ , \tag{17}$$

and

$$s_d = \sum_{j=1}^{n} (d_j - \bar{d})^2/(n-1)^{1/2} \ . \tag{18}$$

Now $\bar{d}$ is an estimate of the bias in the measurements (i.e., relative to the audited value). Assuming the audited data to be unbiased, the existence of a bias in the field data can be checked by the appropriate t-test, i.e.,

$$t_{n-1} = \frac{\bar{d} - 0}{s_d/\sqrt{n}} \ . \tag{19}$$

See Reference 7 for a discussion of the t-test.

If t is significantly large, say greater than the tabulated value of t with n - 1 degrees of freedom, which is exceeded by chance only 5 percent of the time, then the bias is considered to be real, and some check should be made for a possible cause of the bias. If the calculated t is not significantly large, then the bias should be considered zero, and the accuracy of the data is acceptable.

The standard deviation s_d is a function of both the standard deviation of the field measurements and of the audit measurements. Assuming both the field and audited measurements are independent and are obtained using methods whose standard deviations are expected to be the same, then s_d is an estimate of $\sqrt{2}\ \sigma\{B_{wo}\}$. Table 5 contains an example calculation of $\bar{d}$ and s_d starting with the differences for a sample size of n = 7. See the final report on the contract for further information concerning this result.

This standard deviation can then be utilized to check the reasonableness of the assumptions made in section 4.1 concerning $\sigma\{B_{wo}\}$ = 1.2 percent. For example, the estimated calculated standard deviation, s, may be directly checked against the assumed value, $\sigma\{B_{wo}\}$, by using the statistical test procedure

$$\frac{\chi^2}{f} = \frac{s^2}{\sigma^2} \tag{20}$$

where χ^2/f is distributed as a chi-square distribution with $f = n - 1$ degrees of freedom. If χ^2/f is larger than the tabulated value exceeded only 5 percent of the time, then it could be concluded that the test procedure is yielding more variable results than assumed by the specified σ value due to faulty equipment or operational procedure. The values of s_d can be used directly in the test given above, if $\sigma^2\{B_{wo}\}$ is replaced by $2\sigma^2\{B_{wo}\}$, on the assumption that the variance of the field measurements is equal to that for the audited data. Thus,

$$\frac{\chi^2}{f} = \frac{s_d^2}{2\sigma^2\{B_{wo}\}} \quad . \tag{21}$$

The measured values should be reported along with the estimated bias, standard deviation, the number of audits, n, and the total number of field tests, N, sampled ($n \leq N$). If the sample statistics $\bar{d}$ and s_d differ significantly from the population parameters; i.e., $\mu = 0$ and $\sigma\{B_{wo}\} = 1.2$ percent (absolute) for B_{wo}, at the 95 percent confidence level as determined by the t-test and χ^2-test, respectively, they should be identified as being excessive. For example, based on the data of table 5, a measured value of $B_{wo} = 0.15$ (assumed) would be reported with $\bar{d}$ (bias) = + 0.04 percent (absolute) = 0.0004, $s\{B_{wo}\} = s_d/\sqrt{2} = 1.2/\sqrt{2} = 0.85$ percent (absolute) = 0.0085, n = 7, N = 20.

The t-test and χ^2-test described above, and described in further detail in the final report on this contract, are used to check on the bias and standard deviation separately. In order to check on the overall data quality as measured by the percent of measurement deviations outside prescribed limits, it is necessary to use the approach described in the following subsection.

4.3.2 Sampling by Variables

Because the lot size (i.e., the number of tests performed by a team or a laboratory during a particular period, usually a calendar quarter) is small, N = 20, and consequently the sample size is small, of the order of n = 3 to 8, it is important to consider a sampling by variables approach to assess the data quality with respect to prescribed limits; i.e., it is desired to make as much use of the data as possible. In the variables approach, the means and standard deviations of the sample of n audits are

Table 5. Computation of mean difference, $\bar{d}$, and standard deviation of differences, s_d

General Formulas		Specific Example	
$d = (B_{wo} - B'_{wo}) \times 100$		Data	
		d	d^2
d_1	d_1^2	-1.6	2.56
d_2	d_2^2	0.8	0.64
d_3	d_3^2	-0.2	0.04
d_4	d_4^2	-1.0	1.00
d_5	d_5^2	2.1	4.41
d_6	d_6^2	0.5	0.25
d_7	d_7^2	-0.3	0.09
Σd_j	Σd_7^2	+0.30	8.99
$\bar{d} = \frac{\Sigma d_i}{n}$		$\bar{d} = +0.043$	
$s_d^2 = \frac{\Sigma d_i^2 - \frac{(\Sigma d_i)^2}{n}}{(n-1)}$		$s_d^2 = 1.50$	
$s_d = \sqrt{s_d^2}$		$s_d = 1.22$	

used in making a decision concerning the data quality. The optimum value of n is determined as a function of cost in each of the quality assurance documents of this series applicable to pollutant specific methods; i.e., methods 5, 6, 7, etc.

Some background concerning the assumptions and the methodology is repeated below for convenience. However, one is referred to one of a number of publications having information on sampling by variables; e.g., see references 5, 6, 7, 8, 9, and 10. The discussion below will be given in regard to the specific problem herein which has some unique features compared to the usual variable sampling plans.

The difference between the team-measured and audited value of B_{wo} is designated as d_j, and the mean difference over n audits by $\bar{d}$. Equation (17) can be written as

$$\bar{d} = \frac{100 \sum_{j=1}^{n} (B_{wo_j} - B'_{wo_j})}{n} . \qquad (22)$$

Theoretically, B_{wo} and B'_{wo} should be measures of the same moisture content, and their difference should have a mean of zero on the average. In addition, this difference should have a standard deviation equal to $\sqrt{2}$ times that associated with measurements of B_{wo}. Recall from the variance analysis that the difference of two such measurements would have a standard deviation approximately equal to $\sqrt{2} \times 1.2$ percent (absolute) .

Assuming 3 σ limits, the values $-3(1.2\sqrt{2}) = -5.1$ and $+3(1.2\sqrt{3}) = 5.1$ define lower and upper limits, L and U, respectively, outside of which it is desired to control the proportion of differences, d_j. Following the method given in reference 8, a procedure for applying the variables sampling plan is described below. Figures 5 and 6 illustrate examples of satisfactory and unsatisfactory data quality with respect to the prescribed limits L and U.

The variables sampling plan requires the sample mean difference, $\bar{d}$; the standard deviation of these differences, s_d; and a constant, k, which is determined by the value of p, the proportion of the differences outside the limits of L and U. For example, if it is desired to control at 0.10 the probability of not detecting lots with data quality p equal

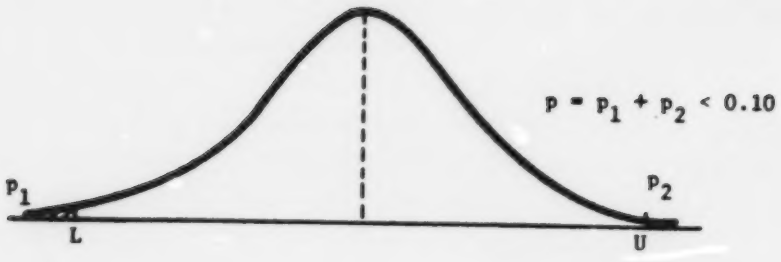

Figure 5. Example illustrating p < 0.20 and satisfactory data quality.

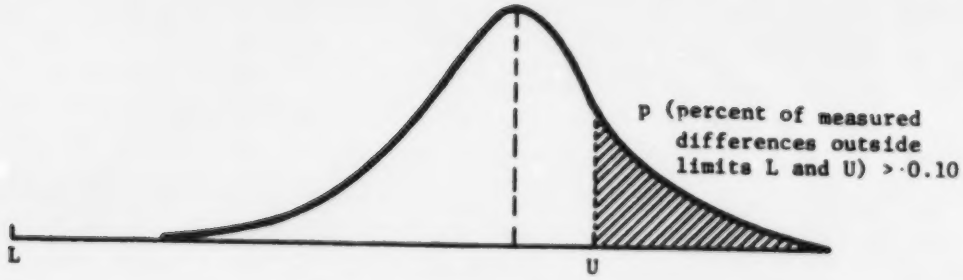

Figure 6. Example illustrating p > 0.20 and unsatisfactory data quality.

to 0.20 (or 20 percent of the individual differences outside L and U) and if the sample size n = 7, then the value of the k can be obtained from table II of reference 8. The values of $\bar{d}$ and s_d are computed in the usual manner; see table 5 for formulas and a specific example. Given the above information, the test procedure is applied and subsequent action is taken in accordance with the following criteria:

1. If <u>both</u> of the following conditions are satisfied:

$$\bar{d} - k\, s_d \geq L = -5.1 \text{ percent (absolute)}$$
$$\bar{d} + k\, s_d \leq U = +5.1 \text{ percent (absolute)}, \tag{23}$$

the individual differences are considered to be consistent with the prescribed data quality limits and no corrective action is required.

2. If one or both of these inequalities is violated, possible deficiencies exist in the measurement process as carried out for that particular lot (group) of field tests. These deficiencies should be identified and corrected before future field tests are performed. Data corrections should be made when possible; i.e., if a quantitative basis is determined for corrections.

Table 6 contains a few selected values of n, p, and k for convenient reference.

Table 6. Sample plan constants, k for P{not detecting a lot with proportion p outside limits L and U} $\leq$ 0.1

Sample Size n	p = 0.2	p = 0.1
3	3.039	4.258
5	1.976	2.742
7	1.721	2.334
10	1.595	2.112
12	1.550	2.045

Using the values of $\bar{d}$ and s_d in table 5, k = 1.721 for a sample size n = 7, and p = 0.20, the test criteria can be checked; i.e.,

$$\bar{d} - k\, s_d = 0.04 - (1.721)(1.22) = -2.06 > L = -5.1 \text{ percent (absolute)}$$

$$\bar{d} + k\, s_d = 0.04 + (1.721)(1.22) = 2.14 < U = 5.1 \text{ percent (absolute)}$$

Therefore, both conditions are satisfied and the lot of N = 20 measurements is consistent with the prescribed quality limits. The plan protects against not detecting lots with 20 percent or more defects (deviations falling outside the designated limits L and U) with a risk of 0.10.

SECTION V REFERENCES

1. Walter S. Smith, and D. James Grove. Stack Sampling Nomographs for Field Estimations. Entropy Environmentalists, Inc., Research Triangle Park, N.C., 1973.

2. Franklin Smith and A. Carl Nelson, Jr. Guidelines for Development of Quality Assurance Programs and Procedures. Final Report, Research Triangle Institute, Contract EPA-Durham 68-02-0598, RTI Project 43U-763, Research Triangle Park, N.C., August 1973.

3. Eugene L. Grant and Richard S. Leavenworth. Statistical Quality Control, 4th ed., St. Louis, Mo.: McGraw-Hill, 1972.

4. David A. Simons. Practical Quality Control. Reading, Mass.: Addison-Wesley, 1970, pp. 131-50.

5. Statistical Research Group, Columbia University. Techniques of Statistical Analysis. C. Eisenhart, M. Hastay, and W.A. Wallis, eds. St. Louis, Mo.: McGraw-Hill, 1947.

6. A. H. Bowker and H. P. Goode. Sampling Inspection by Variables. St. Louis, Mo.: McGraw-Hill, 1952.

7. A. Hald. Statistical Theory with Engineering Applications. New York: John Wiley and Sons, 1952.

8. D. B. Owen. "Variables Sampling Plans Based on the Normal Distribution." Technometrics 9, No. 3 (August 1967).

9. D. B. Owen. "Summary of Recent Work on Variables Acceptance Sampling with Emphasis on Non-normality." Technometrics 11(1969):631-37.

10. Kinji Takogi. "On Designing Unknown Sigma Sampling Plans Based on a Wide Class of Non-normal Distributions." Technometrics 14(1972):669-78.

APPENDIX A

METHOD 4. DETERMINATION OF MOISTURE IN STACK GASES

Figure 4-1. Moisture-sampling train.

(LOCATION)_____ (OPERATOR)_____

(TEST)_____

(DATE)_____

(OPERATOR)_____

(BAROMETRIC PRESSURE)_____

CLOCK TIME	GAS VOLUME THROUGH METER, V_m, ft^3	ΔH/n METER SETTING, $\Delta H/n$	METER TEMPERATURE, t_d, °F

Figure 4-2. Field moisture determination.

and equipped with a filter to remove particulate matter.

2.3 Impingers—Two midget impingers, each with 30 ml. capacity, or equivalent.

2.3 *Ice bath container*—To maintain moisture in impingers.

2.4 Silica gel tube (optional)—To protect pump and dry gas meter.

2.5 Needle valve—To regulate gas flow rate.

2.6 Pump—Leak-free, diaphragm type, or equivalent, to pull gas through train.

2.7 Dry gas meter—To measure to within 1% of the total sample volume.

2.8 Rotameter—To measure a flow range from 0 to 0.1 c.f.m.

2.9 Graduated cylinder—250 ml.

2.10 Barometer—Sufficient to read to within 0.1 inch Hg.

2.11 Pitot tube—Type S, or equivalent, attached to probe so that the sampling flow rate can be regulated proportional to the stack gas velocity when velocity is varying with time or a single traverse is conducted.

3.1 Place exactly 5 ml. distilled water in each impinger. Assemble the apparatus without the probe as shown in Figure 4-1. Leak check by plugging the inlet to the first impinger and drawing a vacuum. Insure that flow through the dry gas meter is less than 1% of the sampling rate.

3.2 Connect the probe and sample at a constant rate of 0.05 c.f.m. or at a rate proportional to the stack gas velocity. Continue sampling until the dry gas meter registers 1 cubic foot or until visible liquid droplets are carried over from the first impinger to the second. Record temperature, pressure, and dry gas meter readings as required by Figure 4-2.

3.3 After collecting the sample, measure the volume increase to the nearest 0.5 ml.

4. Calculations.

4.1 Volume of water vapor collected.

$$V_w = \frac{(V_f - V_i)\rho_w R T_{std}}{P_{std} M_{H_2O}} = 0.0474 \frac{ft^3}{ml}(V_f - V_i) \quad \text{equation 4-1}$$

where:

V_w = Volume of water vapor collected (standard conditions), cu ft

V_f = Final volume of impinger contents, ml

V_i = Initial volume of impinger contents, ml

R = Ideal gas constant, 21.85 inches Hg-cu ft/lb mole-°R.

ρ_w = Density of water, 1 g/ml.

T_{std} = Absolute temperature at standard conditions, 530° R.

P_{std} = Absolute pressure at standard conditions, 29.92 inches Hg.

M_{H_2O} = Molecular weight of water, 18 lb/lb-mole.

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4.2 Gas volume.

$$V_{ms} = V_m \left(\frac{P_m}{P_{std}}\right)\left(\frac{T_{std}}{T_m}\right) = 17.71 \frac{^\circ R}{\text{in. Hg}}\left(\frac{V_m P_m}{T_m}\right) \quad \text{equation 4-2}$$

where:

V_{ms} = Dry gas volume through meter at standard conditions, cu. ft.

V_m = Dry gas volume measured by meter, cu. ft.

P_m = Barometric pressure at the dry gas meter, inches Hg.

P_{std} = Pressure at standard conditions, 29.92 inches Hg.

T_{std} = Absolute temperature at standard conditions, 530° R.

T_m = Absolute temperature at meter (°F + 460), °R.

4.3 Moisture content.

$$B_{ws} = \frac{V_{ws}}{V_{ws} + V_{ms}} + B_{ws} = \frac{V_{ws}}{V_{ws} + V_{ms}} + (0.025) \quad \text{equation 4-3}$$

where:

B_{ws} = Proportion by volume of water vapor in the gas stream, dimensionless.

V_{ws} = Volume of water vapor collected (standard conditions), cu. ft.

V_{ms} = Dry gas volume through meter (standard conditions), cu. ft.

B_{ws} = Approximate volumetric proportion of water vapor in the gas stream leaving the impingers, 0.025.

5. *References.*

Air Pollution Engineering Manual, Danielson, J. A. (ed.). U.S. DHEW, PHS, National Center for Air Pollution Control, Cincinnati, Ohio, PHS Publication No. 999-AP-40, 1967.

Devorkin, Howard, et al., Air Pollution Source Testing Manual. Air Pollution Control District, Los Angeles, Calif., November 1963.

Methods for Determination of Velocity, Volume, Dust and Mist Content of Gases, Western Precipitation Division of Joy Manufacturing Co., Los Angeles, Calif., Bulletin WP-50, 1968.

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APPENDIX B GLOSSARY OF SYMBOLS

This is a glossary of symbols as used in this document. Symbols used and defined in the reference method (appendix A) are not repeated here.

SYMBOL	DEFINITION
N	Lot size; i.e., the number of field tests to be treated as a group
n	Sample size for the quality audit (section IV)
r	Number of replicate analyses per field test
CV{X}	Assumed or known coefficient of variation ($100\ \sigma_X/\mu_X$)
$\sigma\{X\}$	Assumed standard deviation of the parameter X (population standard deviation)
s_X	Computed standard deviation of a finite sample of measurements (sample standard deviation)
μ_X	Assumed mean value of the parameter X (population mean)
$\overline{X}$	Computed average of a finite sample of measurements (sample mean)
$\hat{\tau}_X$	Computed bias of the parameter X for a finite sample (sample bias)
R	Range; i.e., the difference in the largest and smallest values in r replicate analyses
ε_X	Random error associated with the measurement of parameter X.
d_j	The difference in the audit value and the value of B_{wo} arrived at by the field crew for the jth audit
$\overline{d}$	Mean difference between B_{wo} and B'_{wo} for n audits
s_d	Computed standard deviation of difference between B_{wo} and B'_{wo}
p	Percent of measurements outside specified limits L and U
k	Constant used in sampling variables (Section IV)
p{Y}	Probability of event Y occurring

APPENDIX B GLOSSARY OF SYMBOLS—CONTINUED

SYMBOL	DEFINITION
t_{n-1}	Statistic used to determine if the sample bias, $\bar{d}$, is significantly different fr a zero (t-test)
χ^2_f	Statistic used to determine if the sample variance, s^2, is significantly different from the assumed variance, σ^2, of the parent distribution (chi-square test)
L	Lower quality limit used in sampling by variables
U	Upper quality limit used in sampling by variables
CL	Center line of a quality control chart
LCL	Lower control limit of a quality control chart
UCL	Upper control limit of a quality control chart
B_{wo}	Proportion by volume of water vapor in the gas stream as measured by the field team, dimensionless
B'_{wo}	Proportion of volume of water vapor in the gas stream as measured/calculated by the auditor, dimensionless

APPENDIX C GLOSSARY OF TERMS

The following glossary lists and defines the statistical terms as used in this document.

Accuracy	A measure of the error of a process expressed as a comparison between the average of the measured values and the true or accepted value
Bias	The systematic or nonrandom component of measurement error
Lot	A specified number of objects to be treated as a group
Measurement method	A set of procedures for making a measurement
Measurement process	The process of making a measurement including method, personnel, equipment, and environmental conditions
Population	A large number of like objects (i.e., measurements, checks, etc.) from which the true mean and standard deviation can be deduced with a high degree of accuracy
Precision	The degree of variation among measurements (e.g., on a homogeneous material) under controlled conditions, and usually expressed as a standard deviation or, as is done here, as a coefficient of variation
Quality Audit	A management tool for independently assessing data quality
Quality control check	Checks made by the field crew on certain items of equipment and procedures to assure data of good quality
Sample	Objects drawn, usually at random, from the lot for checking or auditing purposes

APPENDIX D CONVERSION FACTORS

Conversion factors for converting the U.S. customary units to the International System of Units (SI)* are given below.

To convert from	To	Multiply by
	Length	
foot	meter (m)	0.3048
inch	meter (m)	0.0254
	Pressure	
inch of mercury (in. of Hg) (32°F)	newton/meter^2 (N/m^2)	3386.389
inch of mercury (in. of Hg) (60°F)	newton/meter^2 (N/m^2)	3376.85
millimeter of mercury (mm Hg) (32°F)	newton/meter^2 (N/m^2)	133.3224
inch of water (in. of H_2O) (29.2°F)	newton/meter^2 (N/m^2)	249.082
inch of water (in. of H_2O) (60°F)	newton/meter^2 (N/m^2)	248.84
	Force	
pound-force (lbf avoirdupois)	newton (N)	4.44822
	Mass	
pound-mass (lbm avoirdupois)	kilogram (kg)	0.4535924
	Temperature	
degree Celsius	kelvin (K)	$t_K = t_c + 273.15$
degree fahrenheit	kelvin (K)	$t_K = (t_F + 459.67)/1.8$
degree rankine	kelvin (K)	$t_K = t_R/1.8$
degree fahrenheit	degree Celsius	$t_C = (t_F - 32)/1.8$
kelvin	degree Celsius	$t_C = t_K - 273.15$
	Velocity	
foot/second (ft/s)	meter/second (m/s)	0.3048
foot/minute (ft/min)	meter/second (m/s)	0.00508
	Volume	
cubic foot (ft^3)	meter^3 (m^3)	0.02832
	Volume/Time	
foot^3/minute (ft^3/min)	meter^3/second (m^3/s)	0.0004719
foot^3/second (ft^3/s)	meter^3/second (m^3/s)	0.02832

*Metric Practice Guide (A Guide to the use of SI, the International Systems of Units), American National Standard Z210.1-1971, American Society for Testing and Materials, ASTM Designation: E380-70, Philadelphia, Pa., 1971.

APPENDIX E SAMPLE AUDIT CALCULATION

A flow chart of the operations involved in an auditing program from first setting desired limits on the data quality to filing the results is given below. Assumed numbers are used and a sample calculation of an audit is performed in the flow chart. Each operation is referred to the section in the text of the report where it is discussed. The information necessary to select an optimum audit level is not given in this document. This information will be given in detail in the final report of this contract and in pollutant specific quality assurance documents of this series.

1. LIMITS FOR DATA QUALITY CAN BE SET BY WHAT IS DESIRED OR FROM THE NATURAL VARIABILITY OF THE METHOD WHEN USED BY TRAINED AND COMPETENT PERSONNEL. FOR THIS EXAMPLE, IT IS ASSUMED THAT $\sigma\{B_{wo}\} = \sigma\{B'_{wo}\}$ AND THAT

   $\sigma\{d\} = \sqrt{\sigma^2\{B_{wo}\} + \sigma^2\{B'_{wo}\}}$. USING $\sigma\{B_{wo}\}$

   = 1.2 FROM THE VARIANCE ANALYSIS

   (subsec. 4.1.1.2), THEN $\sigma\{d\} = \sqrt{(1.2)^2 + (1.2)^2}$ ≃ 1.7. ALSO, THE LIMITS U AND L ARE TAKEN AS ± 3 SIGMA LIMITS: I.E., L = -3(1.7)

   = -5.1 AND U = +5.1.

1 SET DESIRED LOWER AND UPPER LIMITS FOR DATA QUALITY, L AND U

2. BY TEAMS, TYPES OF SOURCES, OR GEOGRAPHY, GROUP FIELD TESTS INTO LOTS (GROUPS) OF ABOUT 20 THAT WILL BE PERFORMED IN A PERIOD OF ONE CALENDAR QUARTER.

2 GROUP FIELD TESTS INTO LOT SIZES OF ABOUT N = 20

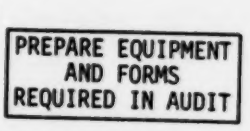
3. SELECT n OF THE N TESTS FOR AUDITING. COMPLETE RANDOMIZATION MAY NOT BE POSSIBLE DUE TO AUDITOR'S SCHEDULE. THE PRIMARY POINT IS THAT THE FIELD TEAM SHOULD NOT KNOW IN ADVANCE THAT THEIR TEST IS TO BE AUDITED. A VALUE OF n = 7 IS USED IN THIS EXAMPLE.

3 RANDOMLY SELECT n OF THE N TESTS FOR AUDITING

4. ASSIGN OR SCHEDULE AN AUDITOR FOR EACH FIELD TEST.

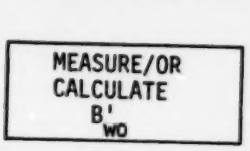
4 ASSIGN/SCHEDULE AUDITOR(S) FOR THE n AUDITS

5. THE AUDITOR OBTAINS APPROPRIATE CALIBRATED EQUIPMENT AND SUPPLIES FOR THE AUDIT (subsec. 4.2).

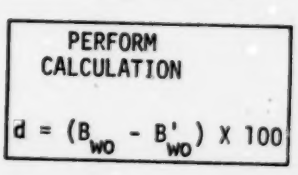


6. OBSERVE THE FIELD TEAM'S PERFORMANCE OF THE FIELD TEST. FILL IN THE AUDITOR'S CHECKLIST (table 4) AND NOTE ANY UNUSUAL CONDITIONS THAT OCCURRED DURING THE TEST.

7. PERFORM AN INDEPENDENT MEASURE OF B'_{WO} OR USING COMBUSTION NOMOGRAPHS CALCULATE A THEORETICAL VALUE OF B'_{WO}



8. CALCULATE d_j FROM EQUATION (15). FOR THIS EXAMPLE, ASSUME THAT IT IS THE FIRST OF SEVEN AUDITS; I.E., $j = 1$. THE FIRST TEAM REPORTED A MEASURED VALUE OF $B_{WO} = 0.15$ PERCENT AND THE AUDIT VALUE WAS $B'_{WO} = 0.166$ PERCENT. THEN FROM EQUATION (15)



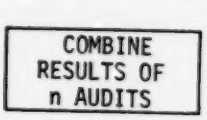
$$d_1 = (0.15 - 0.166) \times 100 = -1.6 \text{ (table 4)}.$$

9. THE AUDITOR'S REPORT SHOULD INCLUDE (1) DATA SHEET FILLED OUT BY THE FIELD TEAM (fig. 3), (2) AUDITOR'S CHECKLIST WITH COMMENTS (table 4), (3) AUDIT DATA SHEET WITH CALCULATIONS, AND (4) A SUMMARY OF THE TEAM'S PERFORMANCE WITH A NUMERICAL RATING (subsec. 4.2.4)

10. THE AUDITOR'S REPORT IS FORWARDED TO THE MANAGER.

MANAGER

11. COLLECT THE AUDITOR'S REPORTS FROM THE n AUDITS OF THE LOT OF N STACKS. IN THIS CASE $n = 7$ AND ASSUMED VALUES FOR THE AUDITS ARE $d_1 = -1.6$, $d_2 = 0.8$, $d_3 = -0.2$, $d_4 = -1.0$, $d_5 = 2.1$, $d_6 = 0.5$, AND $d_7 = -0.3$ (table 4).



12. CALCULATE $\bar{d}$ AND s_d ACCORDING TO THE SAMPLE IN TABLE 4. RESULTS OF THIS SAMPLE CALCULATION SHOW $\bar{d} = +0.043$, AND $s_d = 1.22$ (table 4, subsec. 4.3.2).

12 CALCULATE THE MEAN, $\bar{d}$, AND STANDARD DEVIATION, s_d

13. USE EQUATION (18) TO SEE IF THE BIAS, $\bar{d}$, IS SIGNIFICANTLY DIFFERENT FROM ZERO AT THE 0.05 LEVEL.

13 TEST $\bar{d}$

$$t_6 = \frac{\bar{d}-0}{s_d/\sqrt{7}} = \frac{0.043 - 0}{1.22/\sqrt{7}} = \frac{0.043}{0.46} = 0.093$$

THE t VALUE FOR SIX DEGREES OF FREEDOM AT THE 0.05 LEVEL IS 1.94; HENCE $\bar{d} = 0.043$ IS NOT SIGNIFICANTLY DIFFERENT FROM ZERO AT THE 0.05 LEVEL.

14. USE EQUATION (20) TO SEE IF s_d IS SIGNIFICANTLY LARGER THAN $\sigma\{d\} = 1.7$ AT THE 0.05 LEVEL. IN THIS EXAMPLE $s_d < 1.7$; THEREFORE, NO TEST NEED BE MADE.

14 TEST s_d

15. OBTAIN THE VALUE OF k FROM TABLE 6, FOR $n = 7$ AND $p = 0.2$. THIS VALUE IS 1.721. THEN USE EQUATION (22) TO GET

15 CALCULATE $\bar{d} + k\, s_d$ and $\bar{d} - k\, s_d$

$$\bar{d} + k\, s_d = 0.043 + (1.721)(1.22) = 2.14$$

and

$$\bar{d} - k\, s_d = 0.043 - (1.721)(1.22) = -2.06$$

16. COMPARE THE ABOVE CALCULATIONS WITH LIMITS L AND U (subsec. 4.4.3). FOR THIS EXAMPLE

16 COMPARE (16) WITH L AND U

$$\bar{d} + k\, s_d = 2.14 < U = 5.1$$

$$\bar{d} - k\, s_d = -2.06 > L = -5.1$$

IN THIS CASE BOTH CONDITIONS ARE SATISFIED; THEREFORE, GO TO 18. (IF EITHER OF THE LIMITS HAD BEEN EXCEEDED, CONTINUE TO 17.)

17. STUDY THE AUDIT REPORTS AND FIELD DATA SHEETS TO IDENTIFY CAUSES OF LARGE VARIABILITY. IF QUANTITATIVE DATA ARE AVAILABLE ON SUSPECTED ERROR SOURCES, THE VARIANCE ANALYSIS (subsec. 4.1.1) SHOULD BE WORKED THROUGH WITH THOSE NUMBERS TO VERIFY THE VARIABILITY FOUND IN THE REPORTED DATA. IDENTIFY CORRECTIVE ACTIONS AND NOTIFY THE FIELD TEAMS.

17 MODIFY MEASUREMENT METHOD

18. A COPY OF THE AUDITOR'S REPORT SHOULD BE SENT TO THE RESPECTIVE FIELD TEAM. ALSO, THE DATA ASSESSMENT RESULTS; I.E., THE CALCULATED VALUES OF $\bar{d}$, s_d, AND COMPARISON WITH THE LIMITS L AND U, SHOULD BE FORWARDED TO EACH TEAM INVOLVED IN THE N FIELD TESTS.

18 INFORM FIELD TEAMS OF AUDIT RESULTS

19. THE FIELD DATA WITH AUDIT RESULTS ATTACHED ARE FILED. THE AUDIT DATA SHOULD REMAIN WITH THE FIELD DATA FOR ANY FUTURE USES.

19 FILE AND CIRCULATE OR PUBLISH FIELD DATA